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Laboratory Analysis of Cultural Materials

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Instrumental Techniques in Archeological Research

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This report is designed to be used as a reference guide in applying analytical techniques to archeological research. The report first presents a general approach to instrumental analyses as they are used in archeology and a quick reference (summary chart and matrix) for individual analytical techniques. The remainder of the report is organized by general analytical categories within which a brief description of each analytical technique is presented. An annotated bibliography, organized by technique, is also included. Appendix A lists resources and publications as well as analytical laboratories organized by technique (and cross referenced to an alphabetical listing of addresses and contact persons). Finally, three brief examples of archeological research using some of the techniques discussed in the manual are summarized (Appendices B through D). The examples illustrate how these techniques can be used to solve archeological problems.

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FOREWORD

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INSTRUMENTAL TECHNIQUES IN ARCHEOLOGICAL RESEARCH

1 INTRODUCTION

Background

Federally-funded, -administered, or -licensed construction projects must comply with National Historic Preservation legislation.* This includes not only the preservation and management of prehistoric and historic resources, but, in certain instances, the excavation of cultural materials. These materials must be analyzed and their archeological significance assessed. The U.S. Army Corps of Engineers and other Federal agencies whose projects may impact on cultural resources are required to comply with this legislation.

The general public usually thinks of archeology as fieldwork, and so do agencies that fund excavations. This popular view has led to an overemphasis on collection and description of artifacts and to only limited analysis of the artifacts themselves. The function of archeology is, however, the reconstruction and explanation of cultural change in earlier civilizations. To accomplish this requires a thorough understanding of artifacts: how, when, and where they were made, what they were made of, and the sources of raw materials. Such information provides insight into the technological capabilities of ancient craftsmen, the trade patterns, and the economy of the subject prehistoric society.

Over the past 20 years, the benefits of instrumental analysis of archeological materials have become increasingly apparent. Techniques and instruments borrowed from the fields of chemistry, physics, geology, metallurgy, and ceramic engineering yield quantitative data on archeological artifacts. Early analyses relied primarily on wet chemistry techniques in which samples of artifacts were dissolved into liquid solutions, destroying them in the process. Recently developed techniques, especially those involving x rays or electrical excitation of atoms, require only minute samples, and some are nondestructive, leaving no visible damage on the artifact. Contemporary analyses yield information not only on the major elemental components of artifacts, but also on trace elements, which proves valuable in provenience studies. Other areas benefiting substantially from multielemental analyses are the dating of many types of artifacts and the identification of ceramic technologies, such as the use of tempers and firing conditions.

The difficulty for archeologists today is deciding which analytical method(s) to use in answering their questions. The choice depends on several factors: the question being asked, the amount of money available for analysis, and the equipment available. Optical microscopy is still the first method of analysis in most cases because microscopes are

*Title 16, U.S.C., Section 470 et Seq. (PL 89-665, "National Historic Preservation Act," as amended); Title 16, U.S.C., Section 469 et Seq. (PL 93-291, "Archaeological and Historic Data Preservation Act," as amended); Title 16, U.S.C., Section 470aa-ll, (PL 96-95, "Archaeological Resources Protection Act of 1979"); E.O. 11593, "Protection and Enhancement of the Cultural Environment" (May 13, 1971); Department of Defense (DOD) Directive 6050.1, Environmental Effects in the U.S. of DOD Actions (July 30, 1979).

widely available and are both easy and inexpensive to operate. Microscopic examination yields preliminary answers to such questions as what type of material an artifact is made of and how it was made. This information, combined with stylistic observations made with the naked eye, is usually enough to place the artifact roughly in time and space. The archeologist can then designate specific areas for additional analyses, such as absolute dating, detailed surface examination to determine manufacturing method, or chemical composition analysis to determine source materials.

This research supports the findings reported by the U.S. Congress' Office of Technology Assessment (OTA) in *Technologies for Prehistoric and Historic Preservation*, report number OTA-E-319, September 1986. Specifically, this research supports the OTA finding:

When contracting with private firms or universities for preservation work, the agencies could encourage the use of certain technologies or approaches that have demonstrated a potential for cutting cost and maintaining preservation standards.

Objective

This report is designed to make modern archeometric techniques more accessible to U.S. Army Corps of Engineers (USACE) archeologists. It is intended to be open-ended, allowing for additions as new techniques are developed and refined.

Approach

The process of archeometric analysis is presented, along with specific techniques. Chapter 2 discusses a decision tree that can help the user move from initial analysis by optical microscopy to more sophisticated techniques such as scanning electron microscopy and neutron activation analysis. Since some materials such as pottery can be analyzed profitably using several techniques to yield maximum information, a summary chart and a matrix are provided that outline the advantages and disadvantages of each technique and give cost estimates.

Chapter 3 provides synopses of the analytical methods for ceramics, metal, bone, stone, and other organic and inorganic materials. Advantages and disadvantages are discussed. Each technique is presented with attention to appropriate materials, equipment needed, and sampling procedures. A concluding chapter and an annotated bibliography follow this section.

Appendix A lists organizations, symposia proceedings, and journals that report on instrumental analyses of archeological materials. Laboratories providing archeometric analyses are also listed.

Appendix B presents an example of this type of work based on research currently supported by USACE through the U.S. Army Construction Engineering Research Laboratory (USA-CERL), in conjunction with the Program on Ancient Technologies and Archaeological Materials (ATAM) at the University of Illinois at Urbana-Champaign.

Appendices C and D present further examples of the decision process in archeological analysis, based on analyses of materials from Italy and Ecuador.

2 OVERVIEW OF PROCESS AND TECHNIQUES OF ARCHEOLOGICAL ANALYSIS

The following general discussion and accompanying decision trees are provided to help the user of this report move from preliminary archeological analysis to instrumental archeometric techniques in order to solve problems. For specific examples based on actual studies, see Appendices B through D.

The steps in the process of materials research from problem definition to the evaluation of analytical data are defined below and displayed in a decision tree (Figure 1).

A. Research Model: Ideally, the archeologist follows a research model which complies with the Historic Preservation Act.

B. Research Design: The researcher should have a clear design for his survey and excavation, as well as a realistic appraisal of the finances and manpower available to complete the study.

C. Literature Search: Relevant bibliography should be thoroughly searched at the research design stage in order to assess the potential contribution of the study to the field.

D. Survey and Excavation: Excavation is preceded by test trenches, cores, and surface sampling to assess chronological and spatial limits of the site or sites so that fieldwork can focus on the most important areas.

E. Preliminary Archeological Analysis: A certain amount of analysis is necessary before even beginning to consider archeometric techniques. Types of material, number of samples, archeological context of the samples, and approximate position in time and space of the collection must be identified. In other words, as much information as possible should be collected through traditional typological analyses before trying specialized instrumental techniques to answer specific questions.

F. Definition of Problems for Materials Analysis: Questions should be as specific as possible to employ time and money efficiently. Problems relating to the site itself (intrasite) should be distinguished from those involving several sites or an entire region (intersite). Some questions can be considered on either level. For example, dating can be either relative (intersite) or absolute (intrasite). The composition of an artifact might be an intrasite question, but when provenience of raw materials is considered becomes an intersite question. Both composition and provenience problems usually required elemental analysis.

G. Choice of Analysis: The choice of an analysis procedure depends on C (above) and other factors such as sample size, money available, equipment available, and (in the case of elemental analysis) how many elements are required. For example, an archeologist wanting to know elemental composition of ceramics might choose neutron activation analysis for a large number of sherds and a large suite of elements but choose atomic absorption spectroscopy for a small number of samples and one or two elements.

H. First Level Analysis: In order to conserve time, money, and effort, the simplest form of analysis, such as binocular microscopy, should be tried before more expensive and time-consuming methods. Each analysis may partially answer questions being asked or help narrow the search (see Appendices).

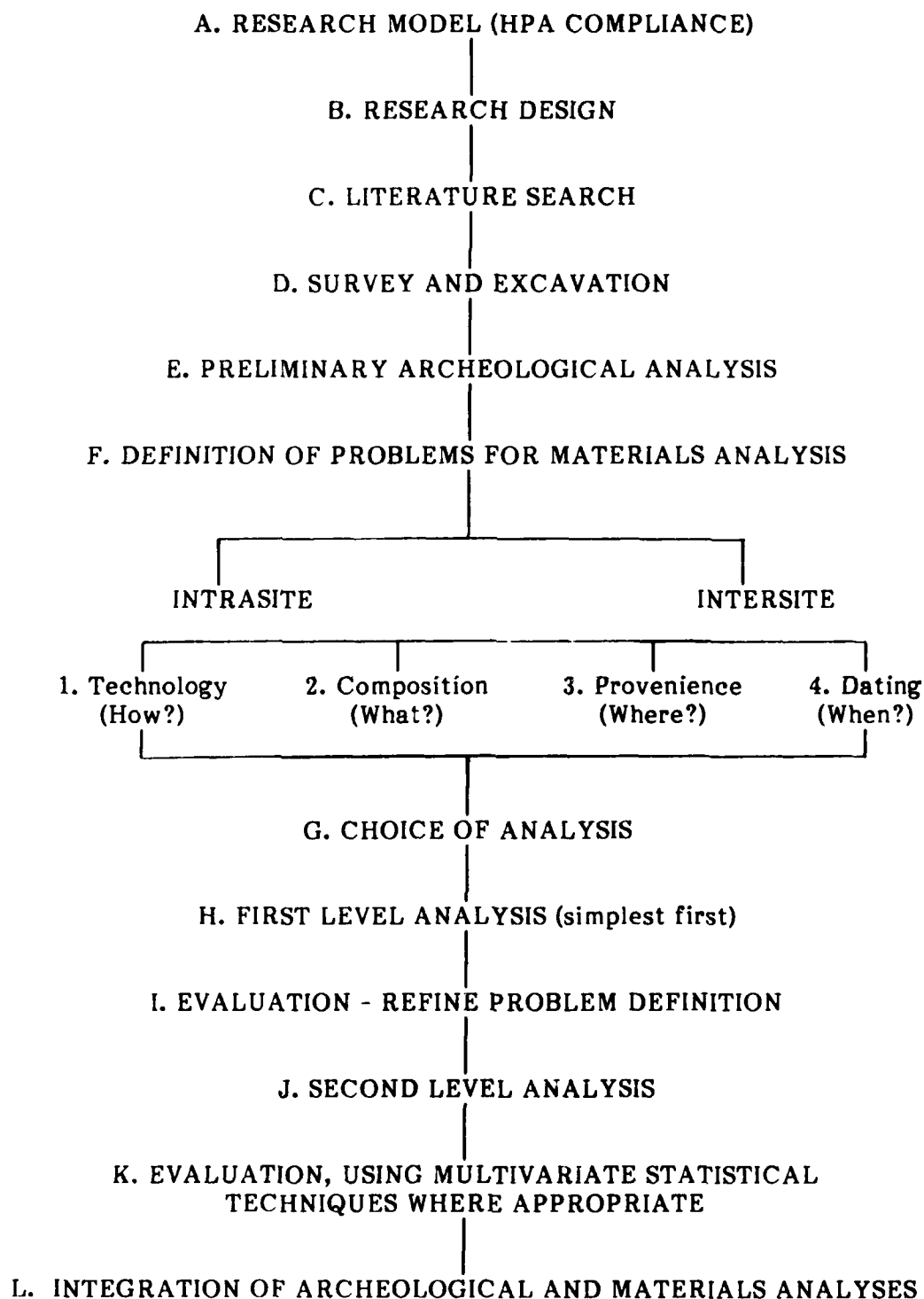
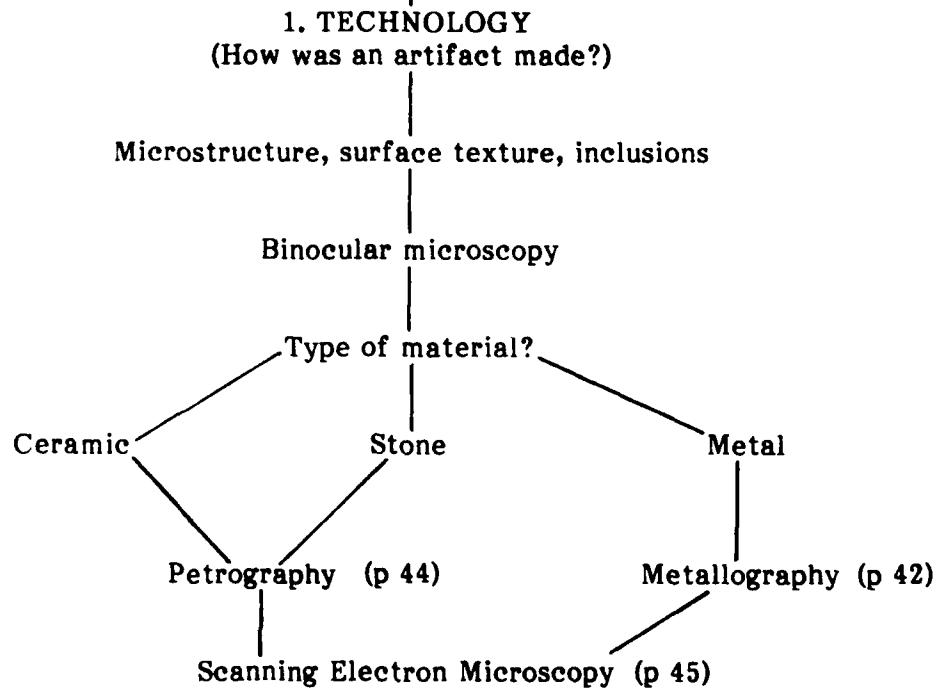
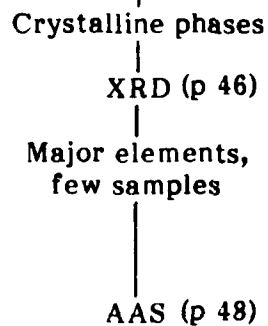


Figure 1. Decision tree for archeological analysis.

SUBTREES FOR (C) DEFINITION OF PROBLEMS



2. COMPOSITION
(What is it made of?)



and

3. PROVENIENCE
(Where did it come from?)

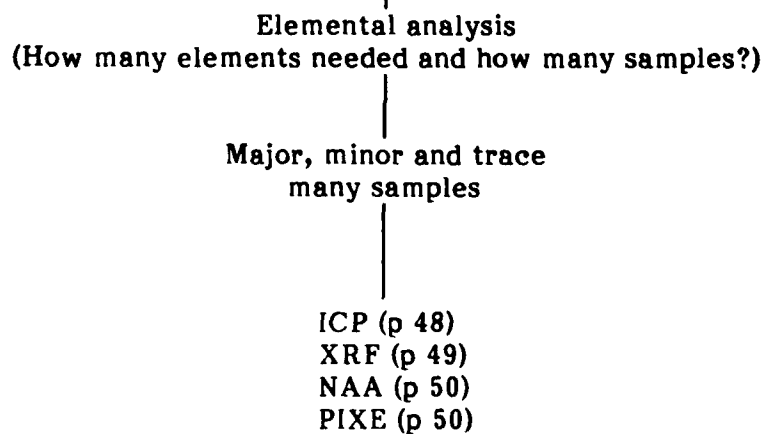


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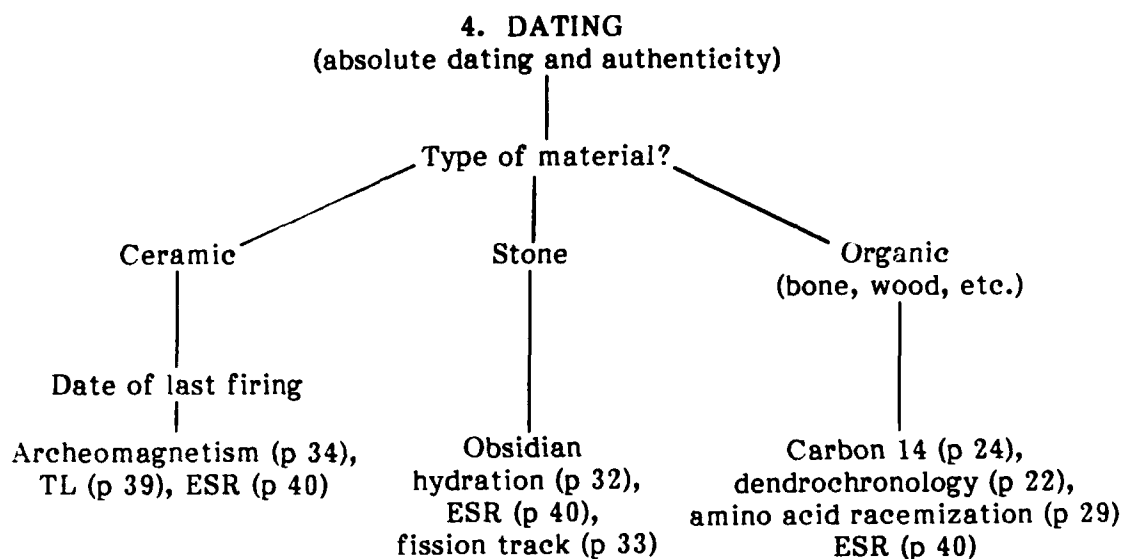


Figure 1. (Cont'd).

I. Evaluation: Results of the first analysis should help determine if further analysis is necessary and if so, which technique is most appropriate.

J. Second Level Analysis: A second type of analysis should yield more information and serve as a cross-check on the result of the first.

K. Evaluation: Final evaluation should take advantage of recently developed hierarchical and nonhierarchical clustering and class modeling techniques which clarify archeological groupings.

L. Integration of Archeological and Materials Analysis: The final step is integrating the knowledge gained from traditional typological methods with the data obtained from instrumental analyses to answer specific problems such as trade patterns and/or locations of materials sources and manufacturing centers.

To help the user choose the appropriate technology for the problem at hand, two tables summarize the analysis techniques described in this report. For each technology Table 1 identifies the equipment needed, the materials for which it is appropriate, the type of sample used, some advantages and disadvantages, and the approximate cost. It is also cross referenced to Appendix A, which lists laboratories that perform these analyses. Table 2 is a matrix that shows which techniques can address which types of problems (composition, provenience, or dating) for which types of samples.

Additional decision trees illustrating the analysis process with *material* as the starting point are displayed in Figures 2 through 4.

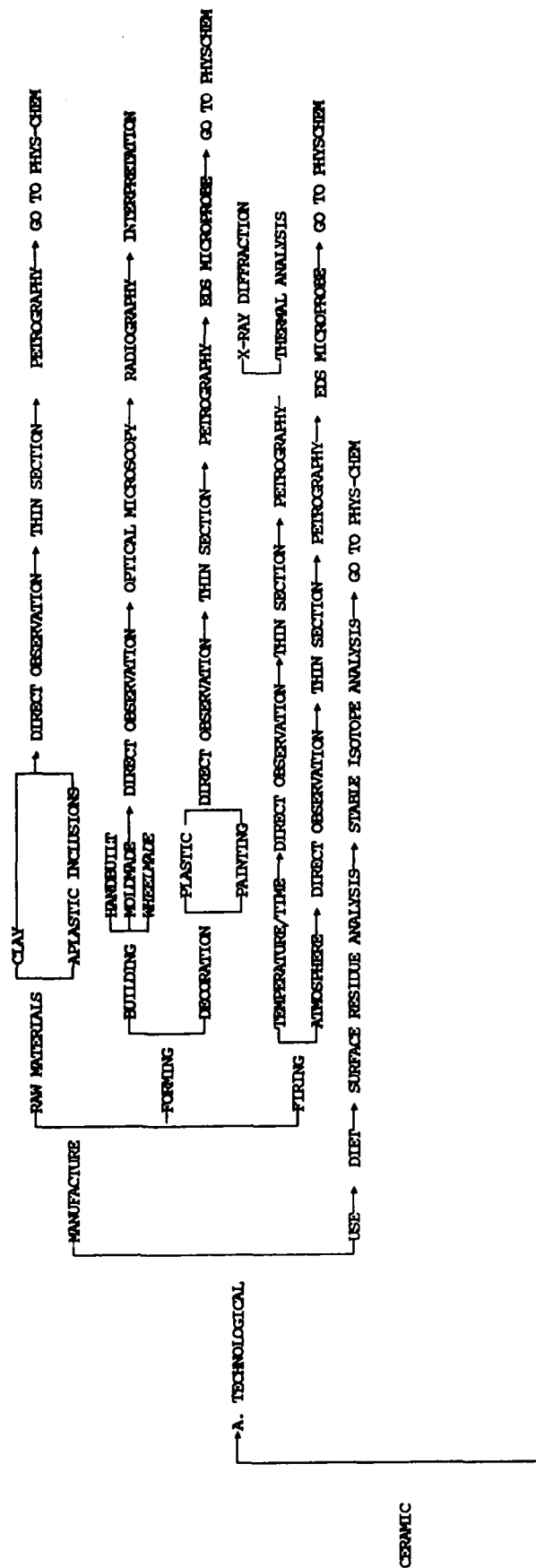


Figure 2. Ceramic decision tree.

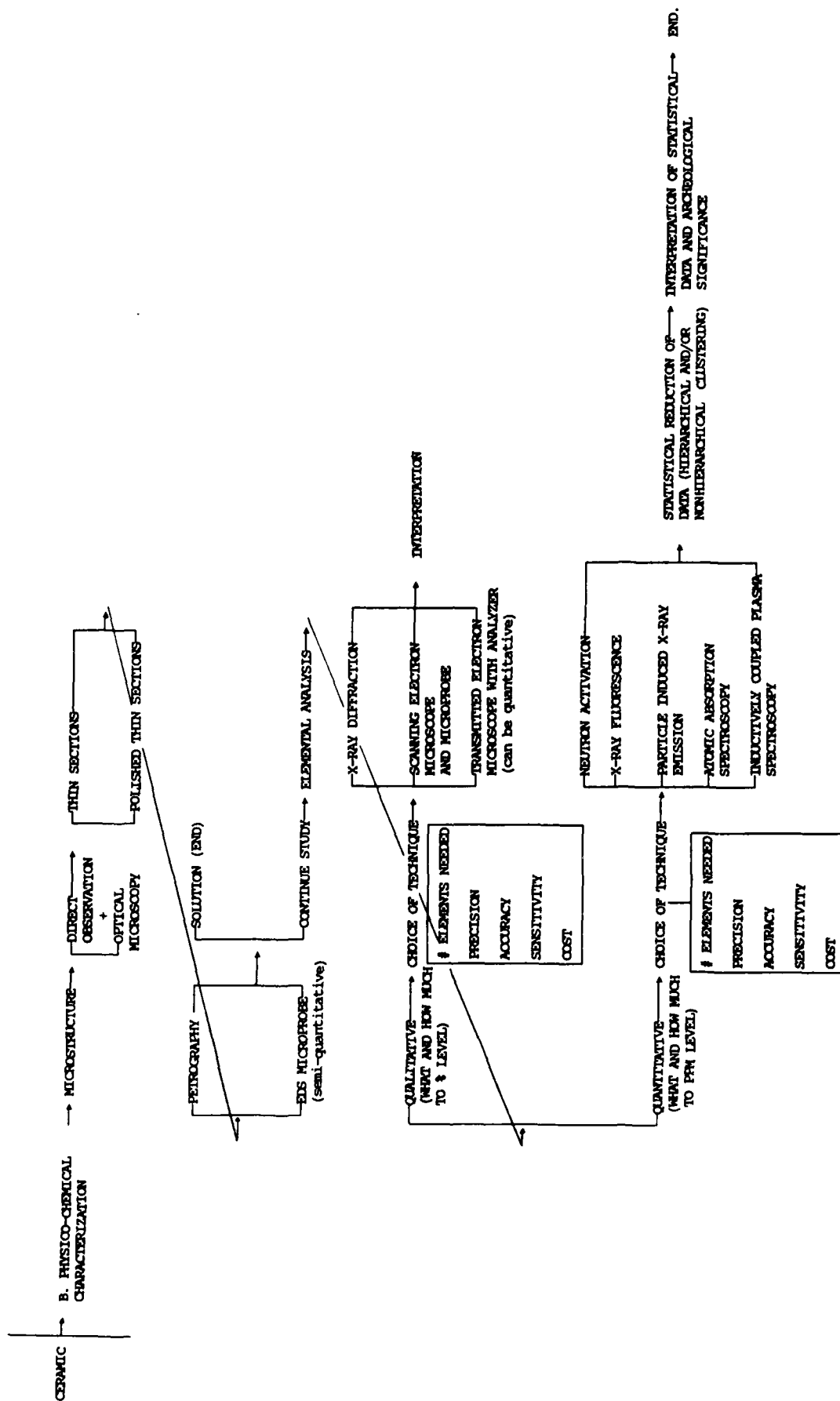


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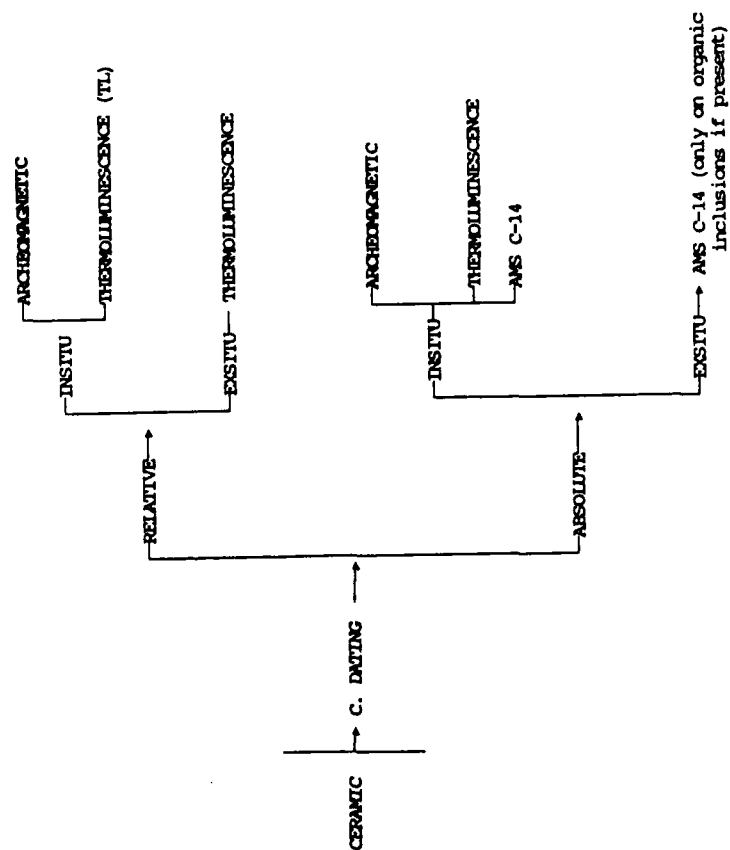


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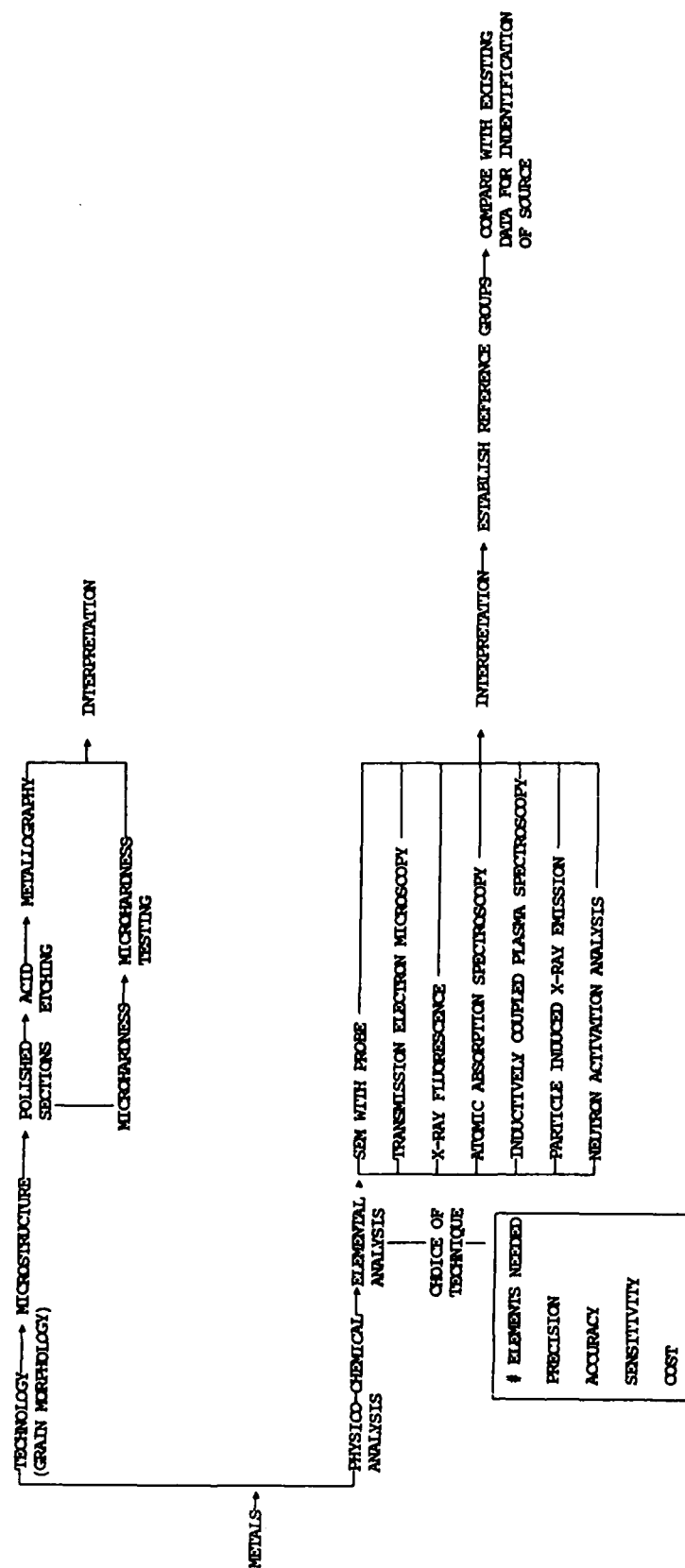
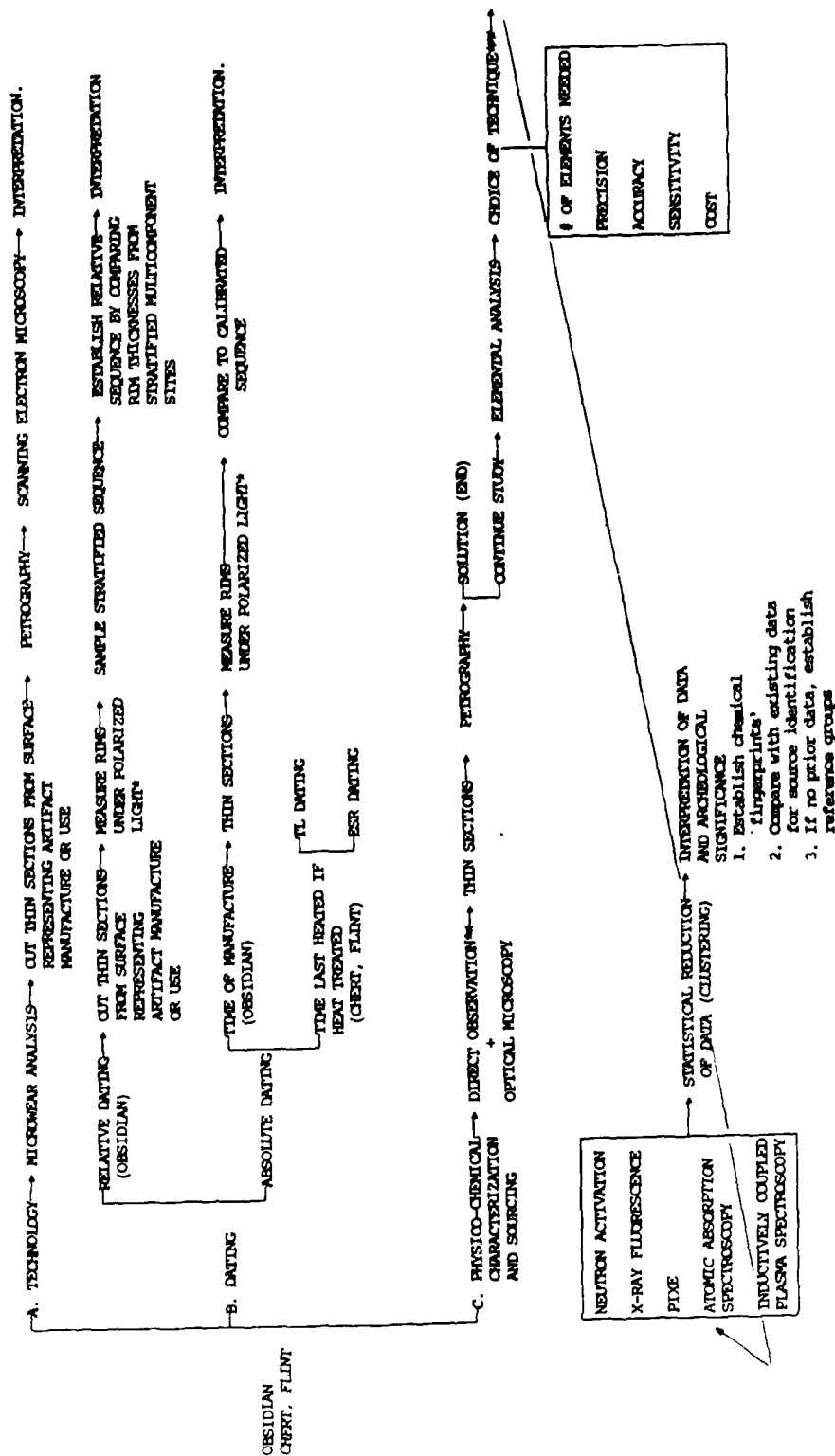


Figure 3. Metals decision tree.



*Some obsidian is so distinctive in appearance (e.g. Pachuca green obsidian) that visual characteristics are sufficient for sourcing. However, in most cases visual criteria are not sufficient and major, minor and trace element analysis is required for identification of obsidian sources.

**If data from previous studies establishing source groups is available, then techniques used reflects sensitivity required to discriminate between potential sources. If no previous studies exist, sampling should include potential sources if available, utilizing the most sensitive quantitative techniques for establishing characteristic suites of elements.

* (OBSIDIAN HYDRATION RIM DATING, P 32)

Figure 4. Obsidian, chert, and flint decision tree.

Table 1
Summary of Archeometric Techniques

B = Bone, C = Ceramic, Ch = Charcoal, G = Glass, H = Humic Acid, L = Lithic, M = Metal,
O = Obsidian, Or = Organic Material, P = Peat, Sh = Shell, So = Soil, W = Wood

Technique	Equipment	Materials	Sampling	Advantages/ Disadvantages	Cost (Commercial lab cost)
Dendro- chronology (App. A: 1)	Calibrated ocular	W	Thin sec- tions of wood cores	Wood only	\$10/sample, minimum 10 samples
Radiocarbon dating (App. A: 2)	Conventional or accelerator mass spectro- meter (AMS)	W, Ch, B, Sh, Or, P, So, H	5-200g (con- ventional) 0.5-5 mg (AMS)	Reliable dates; organic materials only	\$200-400/ sample
Amino acid racemization (App. A: 3)	Gas chroma- tograph	B	Fragments	Needs indepen- dently dated calibrated AAR rate	*
Obsidian hydration (App. A: 6)	Microscope, calibrated ocular	O, G	Thin sections	Can date samples when hydration rate has been calibrated	\$11/sample
Fission track	Electron or petrographic microscope; reactor	O, G	Polished surface required	Covers wide range of dates	*
Archaeo- magnetism (App. A: 4)	Magneto- meter	C, M	Pedestaled cores, oriented in situ	Requires independent calibration	\$160/sample

*Not done commercially.

Table 1 (Cont'd)

**B = Bone, C = Ceramic, Ch = Charcoal, G = Glass, H = Humic Acid, L = Lithic, M = Metal,
O = Obsidian, Or = Organic Material, P = Peat, Sh = Shell, So = Soil, W = Wood**

Technique	Equipment	Materials	Sampling	Advantages/ Disadvantages	Cost (Commercial lab cost)
Thermolumi- nescence (TL) (App. A: 5)	Furnace, pho- tomultiplier tube, electrometer	C, L, M, So	Isolation of grains, 10-50 microns	Dates ceramics and other mater- ials directly. Tedious sample preparation	\$200-300/ sample
Electron spin resonance (ESR)	ESR spectrometer	C, L, B, Sh	150 g, crushed	Still experimental	*
Microscopy (App. A: 7)	Optical microscope	All	Small frags, thin sections	Easy to use, inexpensive	Minimal
Metallography (App. A: 8)	Metallurgical (inverted) microscope	M	Polished sections	Easy to use	Minimal
Petrography	Polarizing optical microscope	C, L, So	Thin sec- tions or crushed samples	Easy to use	Minimal
Scanning electron microscopy (SEM) (App A: 9)	Scanning electron microscope	C, M, B, L, Sh, O, Or, Ch, G, W	Conductive coating for non-metal- lic objects	High power magnification with high resolution	\$50-250/h of instrument time
X-ray Diffraction (XRD) (App. A: 10)	Powder camera, diffractometer	C, M, B, L	Finely ground samples	More infor- mation than petrography	\$100/sample \$300-400/ fine grain analysis

Table 1 (Cont'd)

B = Bone, C = Ceramic, Ch = Charcoal, G = Glass, H = Humic Acid, L = Lithic, M = Metal,
O = Obsidian, Or = Organic Material, P = Peat, Sh = Shell, So = Soil, W = Wood

Technique	Equipment	Materials	Sampling	Advantages/ Disadvantages	Cost (Commercial lab cost)
Emission spectroscopy (ES) (App. A: 11)	Spectrometer	C, M, L	Powdered sample	Superseded by ICP and AAS	\$10-90/ sample, 15- 20 elements
Inductively coupled plasma spectroscopy (ICP) (App. A: 12)	ICP spectrometer	C, M, L	Dissolved sample	30+ elements analyzed at once	\$15-25/ sample, + \$7/element
Atomic absorption spectroscopy (AAS) (App. A: 13)	AA spectrometer; flame atomizer or graphite furnace	C, G, M, L	Dissolved sample	Only 1 element	\$20-25 1st element, \$7-15 each additional element
X-ray fluorescence (XRF) (App. A: 14)	XRF spectrometer	C, G, M, L	0.5 cm ² , clean surface	Nondestructive; only surface probed	\$40/sample, 31 elements
Particle-induced X-ray emission (PIXE) (App. A: 15)	Solid state X-ray detector, electronics	C, G, M, L	Scrapings sufficient	Wider range of elements than XRF, rapid results	\$95-\$500/ sample
Neutron activation analysis (NAA) (App. A: 16)	Nuclear reactor, X-ray detector, electronics	C, G, M, L, B	100 mg, pulverized	Multielemental, trace elements analyzed	\$100-300/ sample, 30+ elements

Table 2
Matrix for Archeometric Techniques

C = Ceramic, G = Glass, M = Metal, S = Stone, B = Bone, W = Wood			
Technology	Composition	Provenience	Dating
Dendrochronology			W
Carbon-14			W,B
Amino Acid Racemization			B
Obsidian Hydration			S,G
Fission Track			S,G
Archeomagnetism	C,M	C,M	C,M
TL			C,M,S
ESR			C,B,S
Optical Microscopy	C,M,S,B	C,M,S,B	C,M,S,B,W
Metallography	M	M	
Petrography	C,S	C,S	
SEM	C,M,S	C,M,S,B	
XRD	C,M,S,B	C,M,S	C,M,S
ES	C,M,S	C,M,S	
ICP	C,M,S	C,M,S	
AAS	C,G,M,S	C,G,M,S	
XRF	C,G,M,S	C,G,M,S,	C,G,M,S
PIXE	C,G,M,S	C,G,M,S	
NAA	C,G,M,S,B	C,G,M,S	

3 INSTRUMENTAL METHODS

The core part of the manual is divided into four sections: (1) dating techniques--analyses used in determination of absolute chronology and in authentication studies; (2) microstructural analysis--the analysis of the microstructure of artifacts by various types of microscopes; (3) phase analysis--the identification of mineral and crystalline phases in artifacts; and (4) elemental analysis--the determination of the chemical composition of artifacts.

Dating Techniques

Dendrochronology

Description. Dendrochronology is a dating method which exploits the relationship between the earth's climate and the growth rate of trees. This method's most significant applications have been as a chronological tool and as a means of reconstructing paleoclimates in the American Southwest, where it was first developed.

In temperate and arctic climates where there are marked growing seasons, trees produce growth rings which represent a single year's growth. In the spring, the growth of the earlywood (large, thin-walled, xylem cells) is generated through the division of the cells of the cambium that lies within the bark. As the growing season progresses, the cells become smaller and the cell walls become thicker, resulting in the production of more compact wood. The boundary between the dense latewood of one season and the porous earlywood of the following year is clearly visible, producing a series of concentric rings. By counting the number of rings from the bark to the pith of a tree (at its trunk), the age of the tree can be determined.

Tree rings can vary in thickness, depending on the climatic and microenvironmental conditions for a given year, by as much as one order of magnitude in successive years. The pattern of ring widths over a span of years can be universal for a given species of trees in a given region. By matching tree ring width patterns of living and dead-fall trees in Arizona, Douglass produced the first dendrochronological sequence, which spanned 500 years.¹ He also produced a "floating" sequence, or relative chronology, by sampling structural timbers of Pueblo Bonito and other pueblo sites of the American Southwest. This sequence could not be tied into the modern sequence due to a hiatus in the occupation of pueblo sites in the Southwest, but it was useful in ordering the sequence of building and occupation of the pueblo sites.

This gap between the pueblo tree ring sequence and the modern tree ring sequence was partially closed by the sampling of structural beams from the Hopi town of Old Oraibi, which has been continuously occupied since A.D. 1260, extending the modern sequence back to almost 800 years before present (B.P.). The gap was completely closed by the excavation of a charred log at the site of Showlow, in east-central Arizona, in 1929. The dendrochronological sequence for the American Southwest now extends back to 322 B.C.

¹S. Fleming, *Dating in Archaeology: A Guide to Scientific Techniques* (St. Martins Press, New York, 1976).

Dendrochronology has been successfully applied in other areas of the world including Alaska, the Great Plains,* Germany, Great Britain, Ireland, Turkey, Japan and Russia.

A number of tree species have been successfully used in dendrochronological studies, e.g., pinon pine, ponderosa pine, Douglas fir, juniper, white fir, Limber pine, bristlecone pine, Sequoia, and even sagebrush.

Method. The method of dendrochronology dating is simple. In living trees, the number of rings from the bark to the pith can be counted to give an accurate age of the tree. However, in archeological wood samples, the bark rarely remains intact; what remains is a pattern of rings, each representing a calendar year of growth, but of unknown age. Working back from the present, a series of patterns of ring widths has been established. The patterns are compared to the tree ring width patterns of ancient samples, and a cross-match is achieved. By counting the number of rings from a tree ring of known age back to the cross-match series of the ancient sample, an absolute date is determined.

When sampling living trees, the investigator removes a pencil-sized core using a coring tool such as the Swedish increment borer. Longer sections are produced using a power-driven core extractor. In instances where the wood or charcoal is waterlogged or fragile, the material is impregnated with a water-miscible wax, polyethylene glycol. These specimens are then razor-cut with a gliding microtome that produces surfaces with good structural resolution. Precision sanding machines are required for the preparation of larger sections. These larger sections are also dusted with chalk to highlight ring structures.

A calibrated magnifying eyepiece is then used to measure ring widths. A number of radial transverses from the center to the exterior of the sample are measured to correct for possible missing rings in some sections.

An alternative method using x-ray microradiographs of wood has been tried in studies of oak beams removed from medieval structures in Europe. The dense latewood portions of the tree rings are represented by light bands on the radiographs. These data are then compared to a master sequence, and cross-match series of tree ring widths are generated, which are then used to derive an absolute date for the sample.

Discussion. As with radiocarbon dating, dendrochronology dates the archeological samples--not necessarily the cultural events which are targeted for dating. In the pueblo examples, arid climatic conditions preserved structural timbers for centuries. In many instances these timbers were reused, producing erroneously ancient dates for the culture. The cultural context of the sample and its history must be taken into consideration when using the sample for dating events at the site.

Dendrochronology has been significantly refined through the efforts of the Laboratory of Tree-Ring Research at the University of Arizona. The application of more precise sequences has made possible detailed studies of the building sequences at individual

*David Stahley (University of Arizona) is compiling a master sequence for Arkansas based on red cedar.

sites, such as Betatakin, in Arizona.² Based on 292 sampled beams from the site, Dean has reconstructed the development of the site from its inception as an intermittently occupied cave in A.D. 1250 to a village of 125 rooms in A.D. 1280. The dating of structural beams at the site gives a room-by-room sequence of construction.

Dendrochronology has also been used to calibrate radiocarbon dating. The bristlecone pine of the White Mountains of east-central California are the oldest living trees in the world. One example is 4,900 years old. These trees grow in arid conditions at elevations of over 10,000 ft. These environmental factors make for short growing seasons which produce well defined growth rings. The dead timbers also are extremely resistant to decay, providing tree ring sequences which have been extended back to the seventh millennium B.C. This long master sequence of tree rings produced major corrections of radiocarbon dates earlier than 3000 B.P. in age (see *Radiocarbon Dating*).

The variation in tree ring width is also a sensitive indicator of paleoclimatic fluctuations. Tree ring width patterns have been used in the American Southwest to identify 12-year drought cycles which date back to the 13th century.

Advantages. Dendrochronological dating produces precise calendar dates which are not plagued by the problems inherent in radiocarbon dating, such as error terms, isotopic fractionation, and correction factors. Tree ring width patterns can be used to reconstruct fluctuations in climate and thus enhance inferences about site and regional sequences. The cost of submitting a sample is \$10 with a \$100 (10 samples or less) minimum required (Laboratory of Tree-Ring Research, University of Arizona). However, not all samples will produce a date. The determination of a date depends on having a sufficient tree ring sequence to produce a match with the master sequence. If a match is possible, the resultant date is considerably less expensive than radiocarbon dating (\$220/sample).

Disadvantages. Dendrochronology has limited application since the method depends on the establishment of master tree ring sequences. These master sequences exist in areas with temperate and arctic climates, where there are marked growing seasons that produce clearly visible rings. Therefore it is only applicable for absolute dating in those regions of North America which have been discussed above. In those areas where master sequences have not been established, tree ring sequences can still be used for relative dating, or "floating" sequences, and for identifying major climatic fluctuations.

Radiocarbon Dating

Description. Radiocarbon dating is the most widely used absolute dating technique in archeology. It can date samples of organic materials ranging in age to 60,000 years B.P. Conventional methods of radiocarbon dating use sample sizes of 1 to 5 g of carbon in gas proportional counters or liquid scintillation counters. Counting intervals can run up to 3 to 4 days. New methods of radiocarbon dating use much smaller samples. Gas minicounters require only 10 to 100 mg of carbon and use extended counting intervals of from 1 to 5 weeks. As many as 15 of these units can be run simultaneously with the aid of microcomputers for counting the data. The very low emission rates and the small sample sizes mean that the best results by gas minicounters are achieved on samples less than 10,000 years old.

²Jeffrey S. Dean, "Aspects of Tsegi Phase Social Organization: A Trial Reconstruction," *Reconstructing Prehistoric Pueblo Societies*, W. A. Longacre, Ed. (University of New Mexico Press, Albuquerque, 1970), pp 140-174.

Samples of 0.5 to 5 mg of carbon can now be dated by the accelerator mass spectrometer (AMS). This new radiocarbon dating method extends the limit of reliable dating to approximately 60,000 years B.P. The use of extremely small samples allows the dating of museum pieces which can provide minute samples without destroying the integrity of the artifact. The high capital costs in setting up a laboratory of this kind will limit the number of institutions which will perform this procedure, but several now exist.

Materials which can be dated must contain carbon which was initially able to exchange with the atmosphere. Commonly used materials are plant and animal remains such as wood, charcoal, bone, shell, and peat. Under certain circumstances, soil and lake and ocean sediments can also be dated as long as the problem of potential contamination is taken into consideration.

Sample weights which will render reliable results are given in Table 3 below. These are given as general weights per sample and can vary depending on the particular conditions under which the sample was deposited.

The principle underlying the dating method is elegantly simple. Carbon exists in three naturally occurring isotopes, ^{12}C , ^{13}C , and ^{14}C . Radioactive carbon is created when cosmic radiation produces neutrons which enter the earth's atmosphere and react with nitrogen to form the carbon isotope ^{14}C .



^{14}C contains 14 neutrons in the nucleus, instead of the usual 12. The extra neutrons render the nucleus unstable and subject to gradual decay. Libby calculated the ^{14}C half-life to be 5,569 years.³ When a neutron leaves the ^{14}C nucleus a radioactive beta particle is emitted. If the initial concentration of radiocarbon is known, the amount of ^{14}C remaining in a sample can be calculated by counting the number of beta emissions per gram of carbon and controlling for the number of background emissions. Using the Libby half-life calculation, the age of the sample can be calculated.

The method depends on five fundamental assumptions:

1. ^{14}C will achieve a uniform global distribution and be taken up in the same proportions by all plant life during photosynthesis.
2. All animal life derived directly or indirectly from plant materials would also be expected to contain the same proportions of ^{14}C .
3. Upon the death of the organism, further ^{14}C uptake or exchange would cease and the trapped ^{14}C would decay exponentially with time.
4. If the specific activity of organic materials has been constant over many thousands of years, then ancient organic material would have exhibited the same specific activity at its death as organic materials at the present time.
5. Then if it were possible to measure the activity of such an ancient sample and compare it to the activity of a modern sample, it would be possible to calculate the time that had elapsed since the ancient sample was in exchange equilibrium with the carbon reservoir.

³W. F. Libby, *Radiocarbon Dating* (University of Chicago Press, Chicago, 1952), p 175.

Criticism of the above assumptions is presented in the discussion section.

Method. Samples are pretreated to remove contaminants. A contaminant is considered to be any carbon which was not an original component of the sample when it was deposited; contamination commonly appears in the form of rootlets and other modern organic material which has entered the sample. The pretreatment process varies from laboratory to laboratory depending on available time, information about possible contamination provided by the archeologists, and available resources. Generally, samples are inspected under low power magnification and all rootlets and other intrusive organic materials are removed. Soil carbonates are removed by the use of an acid wash, where the sample is boiled in 1 to 2 M hydrochloric acid for 1/2 to 1 hour and then washed in distilled water. This treatment also removes other acid-soluble materials. Humic acids are removed in an alkali wash by boiling them in 0.5 to 1 M sodium hydroxide or some other suitable solution. In some cases, badly contaminated wood can be pretreated by preparation of cellulose from the wood sample, but this requires a large sample since 50 to 80 percent of the original material is destroyed.

The porous structure of bone presents serious problems for accurate dating. The best results have been produced when bone collagen is extracted and dated. Amino acids can also be dated if sufficient protein remains in the bone to allow isolation of the amino acids. In cases where the bone is of considerable age, hydroxyproline is isolated and dated by accelerator mass spectrometry. This is a lengthy and expensive process which is generally applied only to very old samples.

Table 3
Recommended Radiocarbon Dating Sample Weights

Sample Material	Conventional (grams)	Minicounter (grams)	AMS (milligrams)
Charcoal	5 - 10	0.1 - 0.5	10 - 100
Wood, dry	10 - 20	0.5 - 1.0	50 - 100
Wood, wet	40 - 80	1 - 2	100 - 200
Bone	100 - 500	10 - 50	500 - 5000
Shell	50 - 100	0.5 - 2	50 - 100
Carbonate	100 - 200	2 - 10	100 - 200
Peat, dry	50 - 100	1 - 3	100 - 200
Peat, wet	100 - 200	3 - 5	200 - 500
Sediment, dry	100 - 200	3 - 5	500 - 5000
Sediment, wet	200 - 500	10 - 50	1000 - 10,000

Source: Richard Gillespie, *Radiocarbon User's Handbook*, Oxford University Committee for Archaeology Monographs No. 3 (Oxford University Press, England, 1984, with corrections added 1986).

The carbon fraction of clean samples is then converted into a suitable compound for measurement of the $^{14}\text{C}/^{12}\text{C}$ ratio. A portion of this compound is analyzed by a stable isotope mass spectrometer to calculate the isotopic fractionation ratio of the sample. For example, certain plants, among them grasses, tend to have higher concentrations of the heavier isotopes of carbon, such as ^{13}C , than is representative of ratios in the standard carbon isotopes (those from Cretaceous carbonate, *Belemnita americana*, from the PD formation of South Carolina [PDB]). Therefore, when dating corn, a grass, the resultant dates are younger than dates from samples of other materials from the same archeological context. Therefore, establishing the ratios of both ^{12}C and ^{13}C to ^{14}C for each sample is necessary. If this isotopic fractionation is not corrected for, dates from samples of different materials are not comparable.

Suitable compounds for measurement vary depending on the laboratory and the method of counting employed. In gas proportional counters and gas minicounters the sample is converted to carbon dioxide, methane, or acetylene gas. In the liquid scintillation method, the sample is converted to acetylene and then converted to benzene by catalytic trimerization. Since each molecule of benzene (C_6H_6) has six carbon atoms, this method produces a compact sample in which the concentration of ^{14}C atoms is increased.

In both the gas proportional and liquid scintillation methods the decay rate of radiocarbon is measured in a standard sample size of gas or liquid. This rate is compared to a modern reference standard and a background standard. The standards represent the initial ^{14}C concentration of all samples and the background radioactivity of the measurement system.

For the AMS method, ^{14}C atoms are detected without waiting for them to decay. Basically, beams of carbon ions from a solid sample are electrostatically accelerated to high energies, and the three isotopes are separated by magnetic fields. The fields separate and clean up the ion beams before they enter the detector, which can identify the ^{14}C ions and discriminate them from other ions of almost equal mass. The principle is that the trajectory of a charged particle moving in a magnetic field is circular, with the radius being proportional to the ratio of mass to charge. Samples and standards are compared at short intervals and the $^{14}\text{C}/^{12}\text{C}$ and $^{13}\text{C}/^{12}\text{C}$ ratios measured.

Radiocarbon age determinations are based on random radioactive decay events and are therefore estimates of the true decay rate values. For this reason radiocarbon dates are given with error terms which represent one standard deviation, which implies a 68 percent probability that the true value for the sample lies within the range of the given error. If the age is bracketed by two standard deviations, the probability increases to 95 percent, and at three standard deviations, it increases to 99 percent. Radiocarbon dates are reported in the following form:

$1000 \pm 75 \text{ RYBP (Laboratory, Sample Number)}$

The 1000 represents the estimated age of the sample, and the 75 represents the error term at one standard deviation (the age is always reported at one standard deviation). RYBP stands for Radiocarbon Years Before Present, which is not to be confused with calendar years. To convert a radiocarbon date to a calendar date, correction factors must be added, including error terms which represent the recalibration of radiocarbon age due to fluctuations in the ^{14}C concentrations in the carbon exchange reservoir over time. The recalibration is based on the radiocarbon dating of wood samples from dendrochronological sequences (see Discussion section, *Dendrochronology*). These factors can add age corrections of up to 700 years depending on the age of the sample.

Finally, the radiocarbon age is followed by an abbreviation for the laboratory which analyzed the sample and the laboratory sample number.

Discussion. Major criticism of the fundamental assumptions of the radiocarbon dating method centers around the constancy of radiocarbon levels over long periods of time. The use of bristlecone pine dendrochronology, which extends back over 9000 years, has demonstrated that there have been major fluctuations in atmospheric ^{14}C concentrations. Radiocarbon dates older than 1500 B.C. can be as much as 700 years too young. Dates younger than 1500 B.C. tend to be in accordance with the tree ring dates. These fluctuations in ^{14}C concentrations appear to be world-wide, due to the rapid mixing of the earth's atmosphere. The causes of these fluctuations are not certain but they may be the result of the combined effects of fluctuations in solar activity, changes in distributions of radiocarbon throughout the various components of the carbon exchange reservoir, and changes in the intensity of the Earth's magnetic field.

Important factors affecting the development of a reliable standard sample are the results of the Industrial Revolution and atmospheric weapons testing. Since 1890, the earth's ^{14}C concentrations have been significantly diluted by the burning of fossil fuel, which has poured stable carbon isotopes into the atmosphere at increased rates. This has resulted in a relative decrease in atmospheric ^{14}C . After 1950, atmospheric nuclear weapons testing produced a rapid increase of atmospheric ^{14}C . These manmade fluctuations in the carbon exchange reservoir have introduced error into the development of a reliable standard for use in radiocarbon laboratories. Corrections for the Industrial and Bomb Effects have been made on the internationally accepted standard, 95 percent pure National Bureau of Standards (NBS) oxalic acid. This is the only standard which should enter into age calculations.

A word of caution: the sample is destroyed by dating. Where large samples are available, they should be divided to allow for future re-dating if problems occur. Also, it is advisable, where possible, to select different materials from the same context to be dated to correct for problems of contamination.

Finally, it must be remembered that the sample is being dated, not the archeological site, feature, or event. Archeologists must make this distinction between the object which is being dated and the cultural event with which the date is associated. Therefore it is extremely important to submit samples which are taken from well defined archeological contexts. It is also important to realize that in some cases samples have been taken from organic material which is considerably older than the target event which the archeologist is attempting to date. Examples of this problem have been encountered in the American Southwest where wooden structural beams have been reused numerous times, and therefore are significantly older than the structures in which they are ultimately found. The usefulness of the date depends upon an understanding of the context from which the sample was recovered.

Advantages. When dealing with time periods extending back 60,000 years B.P., radiocarbon dating is the standard by which other dating methods are judged. Sample collection is relatively simple, and the cost of analysis of a sample by traditional gas or liquid scintillation counting techniques is relatively low--approximately \$220/sample. The method has been used to date hundreds of thousands of samples and has proven to be reliable. Most of the problems with radiocarbon dates come from either poor control of the archeological context of the sample or from contamination which has gone undetected.

The new techniques of radiocarbon dating have reduced the size of the required sample to the point where many of the problems previously encountered in producing adequate samples of datable material have been eliminated.

Disadvantages. Radiocarbon dating can only be applied to organic material, which under certain preservation conditions is difficult to recover. It cannot date inorganic, less perishable materials, such as ceramics, stone, or metal, except under rare circumstances when organic carbon has been incorporated in the artifact during manufacture. The new techniques of radiocarbon dating can be expensive, and few laboratories conduct them. Therefore, only the most important samples can be dated by the AMS method, and the turn-around time on samples can be quite long. The benefits of this dating method, however, greatly outweigh its disadvantages.

Amino Acid Racemization

Description. The age determination limits of the radiocarbon dating method (60,000 to 75,000 years B.P.) have encouraged the development of other dating methods which can fill the gap between the upper limit of radiocarbon dating and the lower limits of the potassium/argon (P/A) dating method. (This report does not discuss P/A dating because there are no New World applications--the technique is not applied to problems in the Holocene.) One of these new methods is amino acid racemization (AAR) dating.* The half-life of aspartic acid, the amino acid used in the AAR dating of bone, is about 15,000 years at 20 °C. This compares with the Libby half-life for radiocarbon of 5,568 years. This longer half-life for AAR allows samples from 5,000 to over 100,000 years B.P. to be dated. This is more useful to Old World archeologists, who work with a much longer archeological sequence than New World archeologists, although AAR dating has been applied to New World human skeletal material of possible Pleistocene age (see Discussion below).

Amino acids exist in two forms, L and D isomers, which are mirror images of each other and can be distinguished by their refractive properties under polarized light. L-isomers bend light to the left, while D-isomers bend light to the right. Amino acids commonly found in living organisms consist almost entirely of L-isomer amino acids. D-isomer amino acids do exist in the biosphere but in extremely small quantities. When an organism dies the L-isomer amino acids convert to D-isomer amino acids by the racemization process at fixed rates, depending on the temperature of the environment in which the organism or fossil is deposited and the half-life of the particular amino acid. This process continues until an eventual equilibrium is reached in the D-L isomer concentrations. Since racemization is a chemical reaction, it is greatly influenced by temperature. Changes of 1 °C can change the racemization rate by 20 percent. Thus the temperature environment in which the fossil has been deposited must be known for the production of accurate dates. A method of estimating the temperature history of a fossil is to calibrate the racemization rate by dating a portion of the fossil bone sample by an independent method, such as radiocarbon dating. An independent date allows the racemization rate to be calibrated; thus other samples from the same site can be dated by the D-L isomer amino acid ratios. However, this method assumes that all other fossils from the site have experienced similar temperature histories.

One of the advantages of AAR dating is that it requires only small samples of bone to produce a datable sample of amino acid. This was particularly important prior to the

*Racemization is the reaction in which an optically active molecule is converted into an optically inactive mixture.

development of gas minicounters and AMS techniques in radiocarbon dating. Since the development of these techniques, sample size has become less of a limitation in radiocarbon dating.

Bada has found that in Southern Africa, calibration of racemization rates based on radiocarbon dates from fossil bone in the 18,000 to 20,000 B.P. time range have been reliable for use in D-L isomer amino acid ratios going back 120,000 B.P.⁴ AAR dating has also produced reliable dates on faunal assemblages in Southern Africa, but its application to human skeletal remains of possible Pleistocene age have produced poor results (see Discussion).

Method. Amino acids are extracted from bone and hydrolyzed, and a small droplet of the resulting hydrolysate is placed at one end of a filter paper strip. When this region of the strip is immersed in a selected solvent, each amino acid is drawn up the paper's length at a characteristic rate as the solvent's front advances by capillary action. Each component is then separated into distinct zones on the paper column with the position determined by the relative solubility of the amino acid in the cellulose-bound water associated with the filter paper. Modifications of this technique include pretreatment of the paper to improve resolution and the use of a glass column instead of paper. Chemical development of the paper's surface (usually with ninhydrin) causes staining related to the amino acid concentrations.

Gas chromatography achieves similar divisions of components as they are carried through a column of porous material coated with an organic liquid. Improvements in resolution for this technique have been achieved by the use of extremely high pressures, around 2,000 atmospheres, in carrier gas streams.

The technique destroys a small portion of the original fossil bone.

Discussion. The application of this technique to the problem of dating human skeletal remains of possible Upper Pleistocene age has underscored the problems inherent with this dating method. A number of human skeletons were recovered from geological contexts in California which suggested that they were of Pleistocene age. Radiocarbon dates on the collagen fraction from these skeletal remains strengthened this possibility. AAR dating of the aspartic acid fraction of the bone collagen produced dates, in some examples, of 70,000 years B.P. These data were then used to strengthen the case that humans had been in the New World for a considerably longer time than was generally accepted.

An inherent problem with the AAR method, however, is the calibration of racemization rates due to the temperature history of the fossils being dated. As Bada has suggested (1985) the surprisingly ancient dates for the skeletal material were based on the calibration rates, which were in turn based on the radiocarbon dates.⁵ Subsequently, since the development of the AMS method of radiocarbon dating which uses very small

⁴J. L. Bada and L. Deems, "Accuracy of Dates Beyond the C-14 Dating Limit Using the Aspartic Acid Racemization Reaction," *Nature*, Vol 225 (1975), pp 218-219.

⁵J. L. Bada, "Aspartic Acid Racemization Ages of California Paleoindian Skeleton," *American Antiquity*, Vol 50, No. 3 (1985), pp 645-647.

samples, Taylor et al.⁶ have redated the skeletal material and produced dates which are all modern (Holocene) in age. The reliability of radiocarbon dates has taken precedence over the AAR dates, and the case for these skeletons being of Pleistocene age has been severely weakened.

Bada has redated the skeletal material using the new dates for calibration, and has produced dates which agree with the Holocene radiocarbon dates.⁷ However, in contrast to the relatively small error terms for the radiocarbon dates, the AAR dates have error terms which are many times greater (in some cases approaching $\pm 2,000$ years on dates of 7,000 B.P.). This large error factor greatly reduces the utility of this method to archeologists working in the New World.

However, an interesting application of D-L isomer amino acid ratios is their use to reconstruct paleotemperatures when, through an independent dating method, the age of the fossil is known. Since the two variables which effect racemization ratios are time and temperature, when the age of the fossil is known, D-L isomer ratios can be used to reconstruct the temperature history of the fossil. This can have some utility in paleoclimate studies, or the study of ocean temperatures as seen through D-L isomer ratios in foraminifera in deep sea cores.⁸

Advantages. The AAR dating method has two advantages. First, the method requires very small samples of bone to produce a date. When this method was developed (the early 1970s) radiocarbon dating techniques still required large samples to produce reliable dates with small error terms.

The second advantage to AAR dating is that it can extend the dating of organic materials back to over 100,000 years B.P. This bridges the gap between radiocarbon and potassium-argon dating techniques.

Disadvantages. The new methods of radiocarbon dating have produced reliable dates with small error terms, using very small samples (see section on Radiocarbon Dating). This has effectively removed the advantage of the sample size required for AAR dating.

The reliability of AAR dates has not been established convincingly since calibration of the racemization rates depends on an alternative independent dating technique to establish the temperature history of the fossil. In cases where the antiquity of the fossil is great, there are no dating techniques which can provide dates for the racemization calibration, and therefore rates must be estimated. This introduces significant error into the AAR dating method.

In conclusion, it seems advisable to avoid this dating technique until the method has been perfected. The extended range of radiocarbon dating with the new techniques have produced dates approaching 75,000 years B.P., which is sufficient for most archeological investigations in the New World.

⁶R. E. Taylor, L. A. Payen, et al., "Major Revision in the Pleistocene Age Assignments for North American Human Skeletons by C-14 Accelerator Mass Spectrometry: None Older than 11,000 C-14 Years B.P.," *American Antiquity*, Vol 50, No. 1 (1985), pp 136-140.

⁷Bada, 1985.

⁸J. L. Bada and R. A. Schroeder, "Amino Acid Racemization Reactions and Their Geochemical Implications," *Naturwissenschaften*, Vol 62 (1975), pp 71-79.

Obsidian Hydration Rim Dating

Description. Obsidian is a naturally occurring glass formed by the rapid cooling of viscous lavas. Because of its hardness and tendency to fracture, producing sharp edges, obsidian has frequently been used to make weapons and tools. A newly exposed surface will hydrate, forming a hydration rim which grows thicker with time. Commonly observed rims range in thickness from less than 1 micron to 20 microns. Measurements of hydration rims on samples of known age are used to calibrate a time versus thickness scale which can be used to date similar obsidian artifacts. Dates obtained by obsidian hydration rim thickness measurements are relative and only as good as the method used for calibration. Obsidian as young as 50 years and as old as half a million years can be dated. In many cases the artifact will have more than one rim thickness, with each thickness reflecting a reshaping of the artifact. Often a hydration rim represents the age of the formation of the obsidian. This technique can also be applied to manmade glasses.

Method. The thickness of the hydration rim is measured by using a petrographic microscope to examine a thin section of obsidian, which is cut perpendicular to the surface of the artifact. Rims are produced naturally as obsidian turns into perlite, which is hydrated glass containing about 3 percent water. The rim is easily detected because its optical properties differ from those of obsidian. When obsidian hydrates, it becomes strained and this strain is visible as birefringence. The contrast between unaltered and hydrated obsidian is sharp, resulting from a very narrow diffusion front.

The general equation used in dating obsidian is:

$$x = kXt^n$$

where x is the thickness of the rim, k is the diffusion coefficient, t is time since exposure of the fresh surface, and n is a constant which is usually equal to $1/2$. When this equation is solved for time, most of the uncertainty concerns the value of the diffusion coefficient k : the rate of diffusion depends exponentially on temperature through an Arrhenius equation with values ranging from 0.36 for the Arctic to 11.0 for the Tropics. More uncertainty in the value of k reflects the fact that different obsidians hydrate at different rates under similar conditions. If the obsidian artifact was in two or more temperature regimes, the resulting date will be incorrect. The best method to determine the value of k is relating hydration rim thicknesses on several artifacts of known but varying ages and then calculating the value for each. If a statistically good value is obtained, it can be applied to similar artifacts found in the same context. The diffusion coefficient can be calculated by studying seasonal temperature changes at the site. However, an error of 1°C in the weighted, average yearly temperature can result in an error of 5 percent in the date. While it might be expected that the relative humidity of the climate would affect the diffusion rate, studies have shown this not to be the case because of the hygroscopic nature of obsidian.

Discussion. Obsidian hydration rim dating has several applications to archeological research. Samples of obsidian tools can be compared to determine which artifacts are of original manufacture and which have been reused. Relative intensity of occupation of a site through time can be measured by graphing the frequency distribution of a large sample of domestic artifacts at different time periods.

Advantages. Hydration rim dating is a simple method, requiring only a petrographic microscope and means for preparing thin sections. Compared to other dating methods, hydration rim dating is both cheap and fast: 100 commercially processed

obsidian dates can be obtained for the same cost as 5 commercially processed radiocarbon dates.

Disadvantages. The method is specific to glass artifacts, either obsidian or synthetic, and temperature variations can invalidate the results.

Fission Track Dating

Description. Fission track dating can be used on any material containing sufficient uranium to produce a high density of tracks. Zircon, obsidian, manmade glasses, tektites, apatite, sphene, and other uranium-bearing phases are the materials most commonly addressed by fission track dating. Archeological samples containing these phases which have been heated to temperatures higher than 700 °C can be dated by this technique with an accuracy of 10 percent. The resulting dates represent the age of the last heating. Fission track dating has been used by archeologists to date material as young as 100 years and by geologists to date materials billions of years old. This method has also been used to date nonfired geologic materials in source area studies.

Method. A small percentage of the naturally occurring ^{238}U nuclei in the artifact decays by spontaneous fission, producing charged particles with high energy. These particles interact with the artifact to generate narrow tunnels called "fission tracks," where the original material is severely disrupted, which average 40 angstroms in diameter and 25 microns in length. These fission tracks are visible with an electron microscope or with a petrographic microscope after a polished surface has been etched. The density of fission tracks depends on the time since the last heating and on the concentration of ^{238}U . All previous fission tracks can be annealed out by rapid heating of the material to 700 °C or by heating to a lower temperature for an extended period of time. Pottery and other fired materials are suitable for dating.

For optical microscopy, the uranium-bearing phase is isolated and embedded in plastic prior to polishing. After polishing, the sample is etched using a procedure suitable for that material. After etching, the concentration of the fission tracks is measured with a petrographic microscope. In order to obtain a meaningful age, the sample must contain enough fission tracks to be statistically valid but not so many that counting becomes impossible. Next the sample is heated to 700 °C to remove all the fission tracks and then repolished. The sample is then exposed to neutron irradiation to induce fission of the rare ^{235}U nuclei, and the fission track concentration is measured again. From the new fission track concentration the uranium concentration can be calculated. The age is calculated from the equation:

$$\frac{P_s}{P_L} = [\exp(\lambda D A) - 1] \frac{\lambda F}{\lambda D} f \quad [\text{Eq 1}]$$

where P_s is the number of spontaneous fission tracks per unit area, P_L is the number of produced tracks per unit volume, λF is the spontaneous-fission decay rate, λD is the total decay rate, and f is the fraction of uranium caused to fission by the irradiation. The value of f is determined by comparison with standards.

Discussion. Fission track dating is particularly useful in studies of stone artifacts which were heated by manmade fires. Stone in contact with high heat can lose its original fission tracks, so that subsequent track accumulation would date from the time of burning. Examples of such material are hearth stones, boiling stones, burnt clay, and fired ceramics.

Advantages. As an elemental dating method, the fission track approach has the merit of covering a wide range of dates, from recent to geological.

Disadvantages. Fission track dating, like neutron activation analysis, requires a neutron source (nuclear reactor) for calibration. The technique is limited to those materials containing sufficient uranium.

Archeomagnetism

Description. Archeomagnetic direction dating exploits the temporal variations in direction and intensity of the earth's magnetic field and the ubiquity of ferromagnetic minerals in the earth's sediments and clays. When ferromagnetic minerals are heated to the Curie temperature--that temperature above which the mineral loses its magnetism--and then cooled, they will reorient to, and retain, the direction of the ambient magnetic field and a measure of its intensity. Measurements of the magnetic direction of this thermal remnant magnetism (TRM) can then be compared to temporal sequences of variation in magnetic direction for the appropriate geographical area (polar curves)--usually areas of 800 to 1600 km diameter. Matches between the magnetic direction of the specimen and the magnetic direction on the polar curve can produce dates accurate to ± 15 to 60 years. This technique can be applied to many burned archeological features, such as hearths, kilns and the walls and floors of burned structures. It is a valuable chronological tool which supplements dendrochronology and radiocarbon dating.

Paleointensity data have the potential to be used in a similar manner. "Paleointensity measurements can be made on unoriented samples of fired clay, thus avoiding the tedious field procedures necessary for archeomagnetic direction dating."⁹ This method, if it is perfected, could be used to directly date ceramic artifacts.

Method. The core of the earth is a molten mass under tremendous pressure. Radioactivity in the interior and cooling at the surface of the planet generate a complex system of electrical currents which in turn generate a magnetic field around the earth. The earth thus behaves like a giant dipole magnet, with north and south poles which differ slightly (about 11.5 degrees) from the north and south poles of the rotation axis. A secondary component of the field, the "nondipole" field, is probably caused by irregularities in the interface between the earth's mantle and core.

Since the earth's mantle rotates more rapidly than the core, the magnetic field orientation or direction changes over time. The relationship of the geographical north pole to the magnetic north pole is thus a measure of the time-dependent direction of the magnetic field. The magnetic poles have been known to reverse positions at different times in geological history.

The intensity of the magnetic field also varies over time, being affected by changes in the tilt of the magnetic dipole axis and variations in the strength of the electrical currents inside or on the surface of the earth's core.

These variations in direction and intensity of the magnetic field over time form the basis of the two types of archeomagnetic dating in use today.

⁹Daniel Wolfman, "Geomagnetic Dating Methods in Archaeology," *Advances in Archaeological Method and Theory*, Michael B. Schaffer, Ed. (Academic Press, New York, 1984), pp 363-458.

Archeomagnetic Direction Dating. The earth's magnetic field can be defined at a given locality by three measurements: the angle of declination, the angle of inclination (or dip), and magnetic intensity. Declination, D , is the angle between magnetic north and geographical ("true") north, which changes from year to year. Over 10 to 20 years, this change can amount to 1 degree or more. Inclination, I , can be defined as the inclination to the horizontal direction made by a freely suspended magnetized needle. This dip varies with latitude from 0 degrees at the magnetic equator to 90 degrees at the magnetic poles. Magnetic field intensity, F , is also latitude dependent, being less than half as strong at the magnetic equator as it is at the magnetic poles.

Archeomagnetic direction dating depends on knowing the secular variations--the changes in D and I over time--of the magnetic field in the geographical area being studied. Calibration curves have been constructed for several parts of the world, for example, the American Southwest and Europe. Dubois¹⁰ has measured TRM directions from samples from the American Southwest which are datable by other means. His polar curves have been recently revised and are now in agreement with dendrochronological dates from the same sites.¹¹ Thellier¹² has produced a secular variation curve for France covering the last two millennia.

The date for an unknown archeological feature at a specific site can be obtained by measuring the direction of the TRM and comparing it to polar curves for that geographical area. The archeological feature must be an undisturbed, burnt area, such as an evenly baked clay floor. Further, there must be some general idea of the feature's age, since secular variation in magnetic direction is sinusoidal in nature, with the same direction corresponding to several different dates.

Clay contains the iron oxides hematite (Fe_2O_3) and magnetite (Fe_3O_4). Raw clay has a magnetism of almost zero because the atomic groupings ("domains") are randomly oriented. When a ferromagnetic mineral is heated above its blocking temperature (Curie temperature) the magnetism of the mineral is disoriented. "The blocking temperature of most of the remnant magnetism in most igneous rocks and fired clays is between the Curie temperature of magnetite (578 °C) and 100 °C below it."¹³ Upon cooling, the domains reorient with a magnetic alignment parallel to the ambient magnetic field at the time of firing. This TRM is essentially permanent unless the object is refired, but is subject to acquisition of secondary magnetic components which can be quite different in direction from that of the primary TRM.

The magnitude of the secondary components, although minor, can result in significantly different age assignments. Therefore, magnetic cleaning of samples is required to remove this contamination.

¹⁰R. L. Dubois, "Secular Variation in the Southwestern United States as Suggested by Archaeometric Studies," in R. F. Fisher, et al., Eds., *Proceedings of the Takesi Nagata Conference* (Goddard Space Flight Center, Greenbelt, MD, 1975), pp 133-144.

¹¹Sternberg, R. S., *Archeomagnetic Secular Variation of Direction and Paleointensity in the American Southwest*, Ph.D dissertation, University of Arizona (University Microfilms, Ann Arbor, 1982).

¹²E. Thellier, "Sur la Direction du Champ Magnetique Terrestre en France, Durant les Deux Dernier Millenaires," *Physics of the Earth and Planetary Interiors*, Vol 24 (1981), pp 89-132.

¹³Wolfman, p 377.

The sampling procedure for archeomagnetic direction dating is precise and time consuming. The Laboratory for Archaeology and History of Art at Oxford has established a list of appropriate clay sample sites ranging from an evenly fired clay floor *in situ* ("good") to poorly fired tiles or brick ovens ("very poor"). Sampling involves carefully extracting between 5 and 20 tiny pillars of burnt clay to fit into a box frame, which is then leveled and topped with plaster of Paris. Although a 2 in. by 2 in. frame has been used, a 4 in. by 4 in. frame is recommended for greater accuracy.¹⁴ The frame is then accurately oriented with respect to both inclination and geographic north using either a Brunton compass or theodolite. If the compass is used, it must be high enough above the sample (about 15 cm) so as not to be influenced by the sample's TRM.

The types of materials which produce the best results are baked clay or earth which is free of large pebbles and is durable enough to remain intact when sampled. Well oxidized material in the red to orange color range which is at least 0.75 cm thick produces satisfactory results.¹⁵

Samples are usually stored for a few weeks at the same bearing orientation at which they were found to remove viscous remnant magnetism (VRM), caused by particles with a low blocking temperature acquiring new magnetic alignments. Other sources of possible error are chemical remnant magnetism (CRM), resulting from chemical and crystalline changes in ferromagnetic minerals below the Curie temperature, or isothermal remnant magnetism (IRM), caused by a strong external field such as that resulting from a lightning strike. These effects can be eliminated in the laboratory by "cleaning" the sample, either by exposure to high temperatures in a progressive heating and cooling sequence, or by exposure to increasingly strong alternating fields (AF demagnetization). Storage in a zero field for a period up to 1 week prior to demagnetization improves the process.

Measurements of remnant intensity and direction are made using one of several types of magnetometers. The parastatic magnetometer, employed by Dubois in the United States, determines physical torque using three suspended magnets. These magnets, and the electric current passing through coils of wire surrounding the instrument, cancel out the effect of the present-day magnetic field. Two other types, ballistic and spinning magnetometers, employ electromagnetic induction. The archeomagnetic sample, which acts as a magnet, induces an electrical voltage when placed near a coil. The voltage is measured by a galvanometer attached to the coil. This voltage is greatest when the sample's field direction is aligned with the plane of the coil. The spinning magnetometer, which induces an alternating voltage by rotating the sample continuously, is the instrument favored by the Research Laboratory for Archaeology and the History of Art at Oxford University and by other Old World archeologists. Recently, a very sensitive cryogenic (low temperature) magnetometer has been used in archeomagnetic studies.¹⁶ The instrument is called a superconducting quantum interference device (SQUID). These instruments operate at very low temperatures, where the electrical resistance of the electromagnet is zero, making possible extremely large currents and hence extremely high magnetic fields. Thus, information can be extracted from very small samples. These magnetometers deliver results almost instantaneously, and as a result archeomagnetic studies have become more numerous and effective.

¹⁴ P. C. Hammond, "Archaeometry and Time: A Review," *Journal of Field Archaeology*, Vol 1 (1974), p 334.

¹⁵ Wolfman.

¹⁶ Wolfman.

The TRM measurement yields the mean values of I (inclination) and D (declination) which define the resultant vector direction for the magnetization. The "Fisher Index," v , is the scatter of readings from several samples taken in the same area. The quantity v_{95} indicates that there is a 95 percent probability that the true magnetization vector lies within a cone defined by v coaxial about the preferred direction. The value for v_{95} is:

$$v_{95} = 0.75 (E_I + E_D \cos I) \quad [\text{Eq 2}]$$

where E_I and E_D are the standard errors for the laboratory measurements of I and D and I is the average value of the inclination.

Paleointensity Dating. Paleointensity dating has the potential to date artifacts no longer *in situ*, such as pottery fragments and terracotta figurines. As with archeomagnetic direction dating, paleointensity dating is dependent on well calibrated regional reference curves.

The method takes advantage of the fact that the relationship between the magnetic moment due to TRM of igneous rock and the intensity of the magnetic field in which the rock is fired is almost linearly proportional up to 1 gauss (the older basic unit of magnetic field intensity, or 1.0×10^{-4} tesla, the new SI unit of magnetic field intensity). "Where this linear relation holds between the natural remnant magnetism (NRM) of a fired-clay archeological specimen and the TRM it acquires when heated to about 700 °C in a laboratory field, F_L , the ancient (or paleo) intensity, F_A , can then be determined by the simple relationship:¹⁷

$$F_A = F_L = \frac{\text{NRM}}{\text{TRM}} \quad [\text{Eq 3}]$$

This relationship does not take into account secondary components of paleointensity caused by VRM, IRM, and CRM. To eliminate the effects of the secondary components Thellier and Thellier¹⁸ developed the Thellier double heating method in which NRM is lost and TRM acquired in a stepwise method of incremental temperature increases between ambient temperature and 700 °C. This method provides a means by which the secondary components of intensity can be identified and removed. However, this method is extremely laborious, which has limited the number of paleointensity studies.

New methods which take advantage of cryogenic magnetometers and micro-processors have reduced the laboratory time needed to process a sample. Recently a cryogenic magnetometer, an oven, and an AF demagnetizer have been housed in the same metal magnetic shield, and the steps in the Thellier method have been automated. This has reduced the laborious aspects of the procedure and increased its speed.

Discussion. Archeomagnetic dating can be used for either relative dating (comparing features at different sites) or absolute dating (comparing TRM measurements with dated values of the geomagnetic field).

¹⁷ Wolfman, p 419.

¹⁸ E. Thellier and O. Thellier, "Sur l'Intensite du Champ Magnetique Terrestre dans le Passe Historique," *Annales de Geophysique*, Vol 15, No. 3 (1959), pp 285-376.

Dating based on archeomagnetic direction measurements has, and will continue to have, the broadest application in archeology. Dating based on paleointensity measurements, although not yet perfected, has the potential to date fired artifacts which are no longer *in situ*, such as ceramics. However, this technique is still in its experimental stages. Nonceramic, portable objects such as coins also have magnetic properties which can be used in paleointensity studies.¹⁹

Current research at Oxford²⁰ may extend the data on geomagnetic field intensity back several thousand years, a study which has implications for radiocarbon dating. Since the variations in cosmic ray flux in the earth's atmosphere over time depend strongly on variations in geomagnetic field strength, it may be possible to calibrate the radiocarbon reference curve beyond the limits of the Bristlecone Pine calibration.

Other applications using TRM data include classifying pottery on the basis of glaze firing temperatures and ordering of architectural parts.²¹

Advantages. Archeomagnetic dating can be used in chronological, provenience, authentication, and classification studies of ceramic, metal, and stone artifacts. It provides a cross-check for other methods such as dendrochronology and radiocarbon dating.

Archeomagnetism is more accurate than any other dating method except dendrochronology. Accurate dating within ± 15 to 60 years is possible with an adequately controlled archeomagnetic polar curve.²²

Disadvantages. Archeomagnetic direction dating has received wide application; however, for it to be successfully applied to chronological problems, it necessitates the construction of accurate, well calibrated polar curves for the particular geographical area in question. These do not exist for much of the world. The construction of polar curves is the first step in the successful application of this technique.

Since secular variation of the magnetic field is sinusoidal in nature, archeomagnetic direction dating depends on a general knowledge of the date of the artifact. This is usually established by another independent dating technique such as radiocarbon or dendrochronological dating. Therefore, the successful application of this technique depends on the presence of some materials which can be dated by an alternative method.

Paleointensity studies are limited by the lack of confidence in the accuracy of regional reference curves. Paleointensity is subject to contamination by secondary components whose effects are poorly understood at present. Further, the sinusoidal nature of intensity versus time curves present the same problems encountered with direction dating techniques, necessitating the use of an independent determination of archeological period.

¹⁹D. H. Tarling, "Archaeomagnetic Properties of Coins," *Archaeometry*, Vol 24, No. 1, pp 76-79.

²⁰E. T. Hall, J. M. Fletcher, and M. F. Barbetti, "Recent Work on Dendrochronology and Archaeomagnetism Dating at Oxford," *Application of Science to Dating of Works of Art, Proceedings of the Seminar September 23-25, 1974* (Museum of Fine Arts, Boston, MA, 1978), pp 73-76.

²¹Hammond, p 335.

²²Wolfman; J. L. Eighmy, *Archaeomagnetism: A Handbook for the Archaeologist*, U.S. Cultural Resource Management Series (Colorado State University, Fort Collins, CO, 1980).

Thermoluminescence Dating

Description. Thermoluminescence (TL) dating can be used to date ceramic material back 200,000 years with an accuracy of 10 percent, but it is more commonly used as an approximate, relative dating technique. It has been used extensively to date ceramics from 300 to 30,000 years old and is the second most common dating technique. It is particularly useful in authentication of pottery. TL has also been applied to burnt stones, cave deposits, and even cast bronzes when enough of the ceramic mold material remains.

Method. When ceramic materials are heated to temperatures between 300 and 500 °C, both thermoluminescent and normal incandescent light are emitted. TL represents the release of stored energy from electrons trapped at imperfections in the crystal lattices. During heating, the trapped electrons return to lower-energy, more stable states and emit light of energy equal to this change. The imperfections are produced mainly by alpha, beta, and gamma radiation from radioactive decay of naturally occurring uranium, thorium, and potassium in the ceramic and the environment.

Heating a ceramic artifact to temperatures greater than 750 °C removes all the radiation damage previously existing in the clay and tempering materials. Most ancient firing processes achieved a temperature of at least 750 °C, so TL observed in a sample (as the damaged areas are eliminated) represents the total dose of radiation to which the ceramic has been exposed since that firing. The intensity of the TL is a function of time since firing, sensitivity to radiation, and annual dose rate. In environments with high annual dose rates, the clay matrix and inclusions reach TL saturation faster than in areas of low annual dose rate. TL saturation is reached when no more energy can be stored in the crystal lattice. The annual dose rate can be calculated from the concentrations of uranium, thorium, and potassium in the ceramic, though knowledge of the burial environment is also important. Sample sensitivity is obtained by measuring the TL of the sample both before and after it has been irradiated with a known radiation dose.

The different TL techniques presently used are the fine-grained technique, the quartz or feldspar inclusion technique, and the zircon inclusion technique.

The majority of radioactive impurities occur in the clay matrix and emit alpha particles as the main type of radiation. Alpha particles travel less than 50 microns in the ceramic and as a result represent internally produced TL trapped primarily in the fine-grained matrix of the material. By using grains less than 8 microns in diameter the fine-grained technique eliminates outside radiation influences.

The inclusion technique examines larger grains: it requires isolation of grains about 90 microns in diameter, with 10 to 20 microns being removed by etching in hydrofluoric acid. The TL in the cores of quartz and to a lesser extent feldspar is the result of gamma and beta radiation produced by the clay matrix with some lesser outside contamination. Since traces of α (alpha) emissions are removed by the etching process, the total TL recorded in these samples is slightly less intense.

Zircons usually contain high concentrations of uranium and thorium, which emit mainly alpha particles. These particles are completely absorbed in the zircon crystal, producing damage, which makes zircon ideal for dating. Slight corrections for gamma and beta radiation are made by comparison with quartz grains.

TL is destructive since several partially crushed samples are required from each sherd. This is necessary because the TL sensitivities can be changed during measurement through exposure to heat. Samples are placed in a furnace designed to heat up to 800 °C rapidly, and TL is detected by a photomultiplier tube. The current produced by the tube is measured with an electrometer which is attached to another recorder, producing a glow curve (light output versus temperature).

Discussion. TL is useful for dating purposes because ceramic artifacts are sometimes the only remains at an archeological site.

Advantages. The advantage of the quartz/feldspar inclusion technique is that the problem of TL saturation is eliminated.

Disadvantages. TL dating of ceramics requires tedious sample preparation and destroys part of the artifact.

The main disadvantage of the fine-grained technique is that TL saturation may occur in environments where there are high concentrations of radioactive elements, giving an incorrect age for the sample. The disadvantages of the quartz or feldspar inclusion technique are the reduced TL and the sensitivity to external radiation effects. Primary disadvantages of the zircon inclusion method are possible saturation effects and the need to isolate zircon grains.

Electron Spin Resonance (ESR)

Introduction. Electron spin resonance (ESR)--also known as electron paramagnetic resonance (EPR)--is an instrumental technique which dates archeological specimens by assessing the amount of radiation damage from soil radioactivity and cosmic rays which has accumulated since the object was made. Thus ESR uses the same specimen information as TL. However, instead of determining the number of trapped electrons (produced by radiation) by thermally bleaching the sample and then measuring the amount of light emitted on recombination, ESR counts the number directly by measuring the amount of incident microwave energy the electrons absorb.

The method has been applied on an experimental basis to ceramics, shells, bone, teeth, flints, hearthstones and a variety of organic materials.

Method. ESR dating utilizes a fundamental property of the electron called "spin," which causes the electron to behave like a small bar magnet. Thus, when placed in a stationary magnetic field, an unpaired electron will align its spin either parallel or antiparallel to the field. Only these two orientations are allowed, and each has a different energy. However, more than two energy levels will result if the electron spin also interacts with the spin of a nearby atom.

The electron can be excited from a low energy state into a higher one if energy exactly equal to the difference between states is supplied. An ESR spectrometer supplies this energy in the form of microwave (electromagnetic) radiation of a specific frequency. The fundamental formula of ESR is thus:

$$h\nu = g\beta B \quad [\text{Eq 4}]$$

where the lefthand side is the energy carried by the quantum of the electromagnetic wave, e.g., the photon, and the righthand side is the difference between the two energy levels for the electron. In this formula, h is Plank's constant (6.626×10^{-34} Joule-sec), ν

is the microwave frequency, β is the Bohr magneton (magnetic moment of the electron, 9.274×10^{-24} joules/tesla), B is the intensity of the magnetic field, and g is the hyperfine splitting constant. The g -value characterizes the local magnetic environment of the electron and is fixed for each type of electron trap. Hence ESR can give both an indication of the number of unpaired electrons, from the decrease in microwave energy, and their distribution among different trapping sites, shown by different values of g . These values correspond to different peaks in the (derivative) spectrum obtained on a CRT or chart recorder as the static magnetic field, B , is slowly varied while the microwave frequency is kept constant.

Specimen preparation for ESR dating can be long, tedious, and finicky, as it must be performed under red light to avoid optical bleaching of the trapped electrons. Crushing, grinding, acid etching, and flotation are often employed, though materials which are rich in the phase containing electron traps, e.g., tooth and bone, are sometimes used unpulverized. The specimen must be approximately 150 grams, and it is loaded into a small-bore quartz tube.

As with TL dating, the most accurate results are obtained if samples of the earth at the excavation site can be analyzed also, but experience has shown that using a constant dose rate of 1 gray/year (0.1 rad/yr) gives results adequate for some purposes (e.g., authentication). Then the age of the specimen is calculated by:

$$\text{Age} = \frac{\text{accumulated dose}}{\text{annual dose rate}} \quad [\text{Eq 5}]$$

Discussion. Although ESR spectroscopy has long been used in both physics and chemistry, its potential as a geological and archeological dating technique has been exploited by only a few laboratories. ESR was first used for geological dating in 1967 by Zeller and coworkers, who showed that a series of Mexican apatites could be correctly dated at 2 to 4 million years B.P.²³ Four years later, as part of a study of radiation centers in sandstone, McMorris used ESR to measure the accumulated radiation exposure of two archeological hearth samples.²⁴ Another application of ESR to geological dating was made by Ikeya²⁵, who used a manganese center in limestone and found a linear relation between signal and dose. These and other archeological applications are given in the bibliography.

Advantages. ESR has the substantial advantage over TL of not destroying the information in the specimen. By contrast, TL measurements involve the freeing of the trapped electrons and the resulting loss of the information their number represents. With ESR, repeated measurements on a single sample are possible, permitting an immediate check of anomalous results or future use of improved equipment.

Another advantage of ESR is that it can be used with partially organic materials--e.g., bone--which would be degraded by the thermal treatment required for TL.

²³E. J. Zeller, P. M. Levy and P. L. Mattern. "Geologic Dating by Electron Spin Resource," *Radioactive Dating and Methods of Low-Level Counting* (International Atomic Energy Agency, Vienna 1967), pp 531-540.

²⁴D. W. McMorris, "Impurity Color Centers in Quartz and Trapped Electron Dating," *Journal of Geophysical Research*, Vol 76 (1971), pp 7875-7887.

²⁵Ikeya, M. and Miki, T., "Electron Spin Resonance Dating of Animal and Human Bones," *Science*, Vol 207 (1980), pp 977-998.

Disadvantages. Unlike TL, which has been applied to archeological materials by numerous laboratories, including commercial testing labs, for many years, ESR is still being developed for this field. Laboratories in Japan and Germany have claimed success with archeological materials, but at present no American commercial testing laboratory can supply a reliable ESR date (this situation could be remedied with more intensive research in the United States). The method is extremely sensitive to paramagnetic impurities in the specimen, light leakage during specimen preparation, grinding of artifacts, saturation of existing traps, and many other experimental problems. It is difficult both to extract a reliable date and to find a laboratory which will perform the test.

Microstructural Analysis

General Microscopy

The hand lens and optical binocular microscope are used in archeological laboratories to assist in visual examination of artifacts. Microscopes are readily available, easy to operate, and relatively inexpensive. Microscopy, which provides valuable information on types of material and techniques of manufacture, should precede more sophisticated and expensive instrumental analyses. Microscopic examination of thin sections and fracture surfaces of artifacts reveals structural detail such as the use of tempering material in pottery, while study of polished sections provides clues about the metallurgical processes employed in making metal objects. Other uses of optical microscopy include examination of microwear on stone tools and butchering marks on bone.

The amount and type of information gleaned from microscopic analysis depends on the magnification and resolving power of the specific microscope and the expertise of the microscopist. The optical binocular microscope, for instance, can be used to determine the size and quantity of tempering materials in ceramic artifacts, but a more detailed image of the surface of the artifact can be obtained using a scanning electron microscope. To determine the total chemical composition of the ceramic object, however, one would be required to use other techniques such as x-ray fluorescence, emission spectroscopy, or neutron activation analysis (see appropriate sections).

The following sections summarize the techniques of microscopy available today, with discussion of sampling procedures, relative expense, and the advantages and disadvantages of each method.

Metallography

Description. Metallography is a form of microscopy which employs light reflected from the polished and etched surface of a sample fragment taken from a metal object. The principle is that although metals are crystalline substances, implying a periodic arrangement of atoms, this periodicity is interrupted and redirected in cast or worked metal objects by the development of small "grains"--particles made up of many molecules. The size and shape of these individual grains, which convey information about the thermomechanical history of the object, can be studied using metallography.

When metals are cold-worked, grain appearance becomes distorted. This distortion is known as "slipping," and appears on etched surfaces as fine parallel lines traversing metal grains. Severe hammering will often flatten the grains. As metal is cold-worked it becomes work-hardened, and eventually will become brittle. In order to retain the malleability of metal while cold-working it, the metal is reheated to the recrystallization temperature, or annealed. The atomic-scale line defects, "dislocations," formed through

the cold-working process disappear, metal grains reform, and the metal becomes soft and malleable again. Since cold-working increases hardness, most prehistoric metal tools and weapons were cold-worked in their final stages of production and left unannealed. Increased hardness of metals is also achieved through alloying, the combining of different metals to produce a solid solution. As alloys cool they form observable and characteristic grain patterns in polished and etched samples. Therefore the grain patterns (morphology) observable on polished and etched samples provide important insights into the process of manufacture and to the function of metal artifacts. For example, since cold-working produces very hard edges, any evidence from the grain showing this process was used suggests the artifact may have been used for cutting. Additional information is conveyed by inclusions and precipitated secondary phases.

Metallography has been employed extensively in European, Asian, African, Middle-Eastern, and American archeology.

Method. The typical sampling procedure involves cutting a small bar from the metal object, imbedding it in a plastic compound, and polishing a surface with progressively finer abrasives. Once the surface is polished, it is etched with acid, which preferentially attacks the grain boundaries. The specimen is then examined with a metallurgical microscope. The microtopographic structure or microstructure can then be observed.

Discussion. The study of artifact function through metallography is particularly effective when the metallographic technique is combined with hardness tests. One standard hardness scale applied to metal is the Birnell scale. Hardness is determined by the depression left in the metal from a small metal ball forced into the object for a standard interval of time at a standard load. The size of the depression is then measured with a micrometer and converted to a Brinell number. For measurements within a limited region, as for a single grain of metal, a microhardness test is used: a diamond point is pressed into the surface, and the size of the impression is measured.

Hardness can be significantly increased through cold-working. The hardness of unworked copper has a Brinell number of about 50, while a cold-drawn copper wire has a hardness of 110. Therefore metallographic evidence for unannealed, cold-worked surfaces can be compared to the hardness of the metal to infer function.

Morse and Gordon²⁶ enlisted a similar strategy to reconstruct manufacturing techniques in the production of copper "money" axes from Mexico. High quality copper sheets were produced to carefully controlled thicknesses through hammering and repeated annealing of copper ingots. The retention of elongated copper arsenide inclusions after annealing is an indication of the total amount of plastic deformation used in the fabrication process. The cutting of axes from the sheets and the hammering of the axe-handle edges was probably done by less skilled craftsmen. Hardness values for the axe-handles are too low to imply utilitarian use of the artifacts.

²⁶B. F. Morse, R. B. Gordon, "Metallographic Examination of Pre-Columbian Mexican Copper and Silver Artifacts from Mitla, Oaxaca (Mexico)," *Archaeomaterials*, Vol 1, No. 1 (1986), pp 57-67.

Lechtman²⁷ compares two approaches to the production of gilded surfaces on silver objects from the Vicus and Chimu cultures of northern Peru, and from Turkey. Here two forms of gilding are defined using metallographic and microprobe analyses. The silver artifacts from Turkey exhibit true gilding with the application of gold foil or leaf onto the silver surface. This is accomplished by burnishing the foil onto roughened surfaces and heating the object to form a zone of interalloyed metal as a bond through atomic diffusion, or by adding mercury to the gold foil and the substrate metal. In Peru, however, surfaces of objects which are made from an alloy of copper, silver, and gold are gold colored through depletion gilding, i.e., the removal of silver and copper from the surface of the object to enrich the relative concentration of gold. Depletion gilding technology is characteristic of techniques used in Colombia and Ecuador rather than Peru, which suggests some form of contact between the three areas.

Therefore, the observed technological approaches to the production of gilded surfaces by use of metallography has provided new insights into the spread of metallurgical techniques in northern South America.

Advantages. The preparation of samples is fairly simple, and a number of samples can be prepared at once. The usefulness of the technique, as for petrography, depends on the experience of the investigator. An experienced metallographer can produce inexpensive and significant results. The samples are also easily adapted to a number of other instrumental techniques which can perform elemental analyses: e.g., energy-dispersive x-ray analysis with a scanning electron microscope, or wavelength-dispersive x-ray analysis with an electron microprobe.

Disadvantages. The correct identification of alloyed metals many times depends on the use of some form of elemental analysis. Metallography alone rarely can identify the composition of impurities or minor amounts of alloying metal in the sample. The process also destroys to a portion of the artifact.

Petrography

Description. Petrographic analysis refers to the microscopic examination of nonmetallic substances to identify their mineral constituents. The method involves the use of a polarizing optical microscope to study how light rays are refracted by the crystalline materials being examined. Reflected light is used for opaque materials and transmitted light for transparent materials. Quite often this form of analysis is coupled with other analyses to quantify the amounts of various minerals in the sample. In archeology, petrography is most commonly used for ceramics, but it can also be applied to soil samples and to stone artifacts.

Method. The principles of petrography derive from optical mineralogy and crystallography. For transparent minerals, light rays are refracted (bent) and partially absorbed as they pass through a crystal with the amount of refraction and absorption depending on the electron and crystal structures. Using polarizer and analyzer attachments, it is possible to measure how much the material rotates the plane of polarization. Because each mineral has a unique set of optical properties, it is often possible to identify what

²⁷H. N. Lechtman, "Ancient Methods of Gilding Silver: Examples from the Old and New Worlds," in *Science and Archaeology*, R. H. Brill, Ed. (Cambridge, MA, MIT Press, 1971), pp 2-30.

mineral or crystal phases are present. Reflected light microscopy uses the same principles as transmitted light except the materials are studied by reflecting the incident light from a highly polished surface.

The use of the transmitting polarizing microscope requires the sample to be thin enough to allow light to pass through the mineral grains. Thus, each sample must be specially prepared prior to analysis. The two common techniques are thin-sectioning and crushing. Preparing a thin section involves grinding a flat surface on the sample, mounting it on a glass slide, and then grinding it to approximately 0.03 mm. This technique has the advantage of providing a permanent record but requires some equipment and time. Thin sections are usually made in batches of 10 to 20 samples with each sample taking an average of 1 hour to prepare. Professional geologists charge by the slide.

Crushing involves reducing the sample to small particles. The mineral particles are then placed on a slide with a refractive liquid for analysis under the microscope. This method is faster than sectioning but does not provide a permanent reference like section, and important mineral relationships are often destroyed during crushing.

Discussion. Petrographic analysis by an experienced optical microscopist can accurately identify minerals present within a sample. Since it is limited to the identification of minerals and to a lesser extent other nonmetallic crystalline materials, petrographic analysis is applied primarily to archeological ceramics.

Advantages. Petrography is cheaper than more sophisticated types of analysis and uses widely available equipment which is easy to operate.

Disadvantages. Processing time, especially for thin sections, inhibits large scale examination of materials. Therefore, petrography is best used for looking at specific properties of the material to address some larger research question such as identifying tempering material and clay sources or distinguishing different ceramic technologies. To be successful in sourcing clay materials, one must know the potential parent materials and show that they are distinct. Very homogeneous clay sources or tempering materials will not be distinguishable by this form of analysis.

Scanning Electron Microscopy (SEM)

Description. With its high resolution and great depth of field, the scanning electron microscope is particularly well suited to the detailed examination of surface texture. SEM is often the logical sequel to binocular microscopy. It has been applied to ceramics, metal, and stone artifacts, as well as organic materials such as wood and bone.

Method. In SEM a small artifact is placed in an evacuated column and then is bombarded with a beam of electrons. Secondary electrons emitted by the sample or primary electrons back-scattered from the sample are then collected and amplified by the instrument. Since the beam strikes only one small region at a time, the entire surface must be scanned in order to produce a complete image. The resulting picture is displayed on a cathode ray tube; this image can be photographed or captured for computerized image processing.

Resolution of down to 100 Å with depth of field several hundred times that of optical microscopy can be achieved with SEM. The result is an easily readable picture showing surface topography, similar to that of much larger objects viewed with the naked eye.

The SEM technique is nondestructive in that small objects or fragments can be studied directly. Metallic artifacts can be processed without special preparation, but nonmetallic artifacts must be coated with a thin conducting film such as carbon or gold.

Discussion. SEM has been most extensively used in ceramic analysis, where a close-up of pastes and tempering allows discrimination among various manufacturing techniques. The technique is useful for determining patterns of microwear on stone artifacts and manufacturing technologies for metal artifacts. SEM has also been applied to biological remains such as bone, dental tissue, and hair, and it has contributed to the study of prehistoric subsistence through examination of coprolites and charred plant remains.

SEM gives substantially more topographical information than can be obtained with a binocular microscope. When combined with energy dispersive x-ray fluorescence analysis, the usefulness of the technique is extended even further. For higher resolution and revelation of internal structure, Transmission Electron Microscopy (TEM) is needed.

Advantages. The chief advantages of scanning electron microscopy are: (1) the technique provides both higher magnification and greater depth of field than a binocular microscope, (2) it can be applied to a wide variety of archeological materials, and (3) it is nondestructive to small artifacts or fragments. Reanalysis of the materials is thus possible and provides an opportunity to check the reliability of observations.

Disadvantages. SEM reveals only surface structure; therefore it is not an appropriate method for analysis of internal structure of an artifact. It is more costly in terms of time and money than binocular microscopy and requires specialized equipment and experienced operators.

Phase Analysis: X-Ray Diffraction (XRD)

Description

X-ray diffraction (XRD) is used to identify minerals and other crystalline phases found in archeological samples. The technique identifies mineral particles too small for analysis by petrography. XRD has proven especially useful in the study of ceramics. As the development of crystalline phases in ceramics depends on the firing temperature, XRD results can bracket this important parameter. The technique is a valuable tool in provenience studies of pottery, because sometimes the XRD results can associate raw materials with the finished products. This type of analysis has also been used to study the mineral phases in bone, stone, and metal artifacts as well as paints and other coatings.

Method

Archeologists usually use a powder camera or a diffractometer for x-ray diffraction analysis. Both instruments utilize the diffraction of monochromatic x rays by crystalline materials. The atoms in crystalline solids (both natural minerals and man-made materials) scatter x rays which interfere constructively--i.e., diffract--at specific angles which are related to the spacing between planes of atoms in the crystal. Every crystalline material produces a diffraction pattern which can be used like a fingerprint to help identify the material, though knowledge of the chemical elements present is often necessary as well.

A powder camera and a diffractometer both use an x-ray tube and a filter to produce a monochromatic x-ray beam. In a powder camera, a small amount of finely ground sample is placed on a spindle and rotated inside a disc-shaped camera which contains a photographic film plate. The x-ray beam strikes the rotating sample and diffraction occurs. From the film record, the angles of diffraction and hence the spacing between atomic planes can be calculated.

The diffractometer also uses a finely ground sample, which is placed in a flat sample holder. The beam strikes the sample and the diffracted x rays are detected by a proportional counter or solid state detector. The corresponding diffraction pattern, intensity versus angle of diffraction, is recorded by a strip chart recorder. The diffraction angle, θ , is converted to interatomic spacing (d-spacing) by the Bragg equation

$$n\lambda = d \sin\theta \quad [\text{Eq 6}]$$

Tables can be used to convert between the two, although automated diffractometers which print out the result are now common. Identification is made by matching the d-spacings and relative intensities of the unknown with known values found in the American Society of Testing and Materials (ASTM) powder diffraction index, now available on microfiche and in computer software.

A diffraction pattern can be obtained in a few minutes with a computer controlled, automated diffractometer; a powder pattern from a minute amount of specimen may take many hours. Identification of the pattern may take a few minutes for expected common materials or several days if many materials are present and/or if the materials are rare. Clay minerals often require additional analyses to check the identification.

Discussion

XRD is excellent for qualitative identification of mineral phases but less reliable for quantitative measurement, as different samples of the same mineral can yield different scattered intensities.

Advantages

An advantage of XRD is that it can be applied to a wide variety of artifacts. The technique is more specific than petrography, providing a way to identify crystalline material which often cannot be identified by any other method. XRD also serves as a necessary backup for uncertain petrographic analyses.

Disadvantages

The major drawback with XRD, as with many other methods, is that a sample must be taken from the object and powdered.

Elemental Analysis

Spectroscopy

Description. Spectroscopy, or spectrographic analysis, is one of several elemental analytical methods used to determine the chemical composition of an artifact. The technique involves measuring the emission or absorption of radiant energy by a sample of matter.

Emission spectroscopy (ES), also referred to as optical emission spectroscopy, has long been used to determine minor and trace elements in geological materials and for metallurgical quality control. The most common application of ES for archeological materials is to ceramics. ES is particularly useful in distinguishing clay bodies which appear to be identical microscopically. The method identifies chemical elements by dispersing the light emitted by a thermally excited specimen and measuring its wavelengths and intensities. There are several types of emission spectroscopy, including arc and spark methods, but these are now largely superseded by inductively coupled plasma spectroscopy (ICP).

ICP is useful for analysis of mineral phases, soils, flint and obsidian artifacts, ceramics, and metal. The technique is a type of multielemental analysis, since it can determine from 1 to 65 elements simultaneously.

Atomic absorption spectroscopy (AAS) measures radiation absorbed by atoms in a sample. AAS can be used to determine over 65 major, minor, and trace elements in materials such as ceramics, metals, and stone. Results are particularly useful in provenience studies.

X-ray fluorescence spectroscopy will be considered in a separate section.

Methods. In ES (arc and spark forms), atoms are put into excited higher states of energy using electrical current. The atoms quickly return to their normal (ground) energy states and in the process emit light. Since the wavelength of light emitted by each kind of atom is unique, elements can be identified by the spectrum of light emitted by an excited sample. The light spectra are recorded either by a spectrograph with a photographic plate or film or by a multichamber spectrometer which employs photoelectric detectors. Sample preparation requires that a small portion of the artifact be destroyed by grinding it to a fine powder and mixing it with graphite.

In ICP, an inductively coupled plasma torch replaces the electrical discharge as the source of excitation. ICP measures the intensity of radiation emitted by an element for a specific wavelength. A solution of the sample is aspirated into a stream of ionized gas, which excites the atoms. The concentration of an element is indicated by the intensity of the radiation spectrum emitted by the excited atoms.

In AAS, molecules are dissociated into their constituent atoms, and the radiant energy absorbed by the atoms is measured. The spectrometer consists of a radiant light source (a hollow cathode lamp), a cell containing the sample in solution, a flame source or a graphite furnace, a monochromator, a detector, and a recorder. The solution is aspirated into the flame so that the mist contains neutral atoms of the elements present. Each element absorbs radiation of a characteristic spectral wavelength, isolated by the monochromator (a diffraction grating). The detector then transmits the output to the recorder. The concentration of the element is proportional to the difference between the intensity of unabsorbed radiation and the intensity of radiation after absorption. AAS is a highly sensitive and precise technique, requiring great care in sample preparation. All of the sample must be dissolved, and contamination must be avoided. Only about 10 mg of material is needed, although more may be required if many elements are analyzed. The sensitivity of AAS can be further enhanced by employing an electrothermal atomizer instead of the flame atomizer. Smaller samples, including in some cases solid samples, can be used with this attachment.

Discussion. Although the arc and spark forms of emission spectroscopy are still performed in some laboratories, ICP and AAS are the usual techniques currently applied

to archeological materials. The choice of method used depends on several factors: the number of samples to be run, the number of elements to be analyzed, access to different types of instruments, and cost.

If arc emission spectroscopy is used, only about 15 samples can be run per day. Approximate cost for analysis of 15 samples for 15 to 20 elements would be about \$150 (\$10 per sample). In general, an analysis of a small number of elements and a small number of samples would cost about the same with either AAS or ICP. For example, to run a batch of 50 potsherds for 5 elements would cost approximately \$15 per sample per element using either technique. However, the cost of running samples for a large number of elements goes up sharply in AAS since elements are analyzed one at a time. ICP is more appropriate for multielemental analysis of a large number of samples and has the potential for generating much more information. ICP is also slightly superior to AAS in picking up trace elements when the graphite furnace is not employed.

Advantages. ICP is capable of accurately measuring trace elements such as zircon, zinc, manganese, iron, barium, calcium, and vanadium.

AAS has the advantages of versatility, good sensitivity for many elements, and fast results.

Disadvantages. The primary disadvantage of ICP, as with ES, is that it destroys the sample, although the sample is dissolved in a liquid instead of ground into a powder.

The primary disadvantages of AAS are that it is costly to perform, destructive to the artifact sample, and requires elements to be analyzed successively rather than simultaneously. Also, AAS cannot find carbon, nitrogen, oxygen, or hydrogen, but it can determine the heavier elements in organic materials.

X-Ray Fluorescence (XRF)

Description. X-ray fluorescence is a type of elemental analysis particularly useful in provenience and authenticity studies. Although it has been applied to ceramics, it has proved especially successful in analysis of gold, silver, and copper objects; coins; glass; and obsidian. For example, XRF has been used to correlate obsidian artifacts with source areas by measuring the concentration of trace elements. XRF analysis of coins provides data on relative chronology, provenience, and minting technologies.

Method. XRF is actually a form of spectroscopy, employing either wavelength dispersive or energy-dispersive spectrometers. A primary radioactive source or a standard x-ray generator is used to excite the atoms of the sample. The sample emits fluorescent radiation, with atoms of a particular element emitting x-ray radiation of a characteristic wavelength. The instrumentation is completed by either an energy-dispersive semiconductor detector, which converts the energy carried by the x rays into electrical signals, or by a wavelength-dispersive system which determines the wavelength of the x rays, and equipment for amplification and measurement of the electrical output. After the elements are identified by the energy or wavelength or the emitted radiation, quantitative analysis is performed by comparing the intensities of the x rays produced with those obtained from known standards.

This method of analysis is nondestructive but does require a clean and uncorroded surface for good results. An area of 0.5 cm^2 is usually analyzed with an effective depth varying from a few microns to several tens of microns because of x-ray absorption in the sample volume.

Discussion. If care is taken with sample preparation, x-ray fluorescence is as accurate as neutron activation or atomic absorption. For an element to be detected it should be present in concentrations greater than 10 to 100 ppm, depending on the element. For major elements reproducibility is 5 to 10 percent, and for trace elements reproducibility is 30 percent. XRF analyzes elements from atomic number $Z = 11$ upward.

Advantages. XRF is a nondestructive technique, which produces rapid results for a large number of elements.

Disadvantages. Only the near surface is probed. This limitation presents a problem if the material has been coated--for example, a copper coin dipped in silver or a ceramic vessel covered with a glaze--or if the surface has been altered by the environment. However, if only the coating is to be analyzed, XRF is a suitable method of analysis.

Particle Induced X-ray Emission (PIXE)

Description. PIXE is a form of energy dispersive x-ray fluorescence using charged particles instead of photons to induce x-ray emission. It is a relatively rapid technique which can be applied to analysis of ceramics and other materials. PIXE is extremely sensitive, identifying elements in concentrations ranging down to 1 ppm.

Method. The principle is the same as for XRF, in that an inner orbital electron is knocked out of the atom by the incoming charged particle. Either protons or alpha particles are used.

Sample preparation is simple, requiring only untreated scrapings from a vase or sherd using a commonly available tungsten-carbide tipped tool. Preparation takes less than five minutes per sample and analysis only slightly longer.

Advantages. The primary advantage of PIXE is that it has more even excitation across the periodic table than XRF. Photon excited XRF has a sharply decreasing sensitivity so that it is necessary to bombard with three different energy photons to cover the whole range of elements thoroughly. PIXE is better able to cover a wider range of elements in a single analytical run.

Disadvantages. The primary disadvantage of PIXE is that it is less able to analyze below the surface of an object than photon XRF, because the penetrability of a proton or alpha particle is less than that of a photon. Neither technique can penetrate below corroded surfaces unless the sample is drilled, etched, or scraped down to the lower layer.

Another disadvantage is that it requires a high energy accelerator and thus can be performed only in laboratories so equipped.

Neutron Activation Analysis (NAA)

Description. Neutron activation analysis (NAA) is used to perform multielemental analysis on a suite of samples. Commonly analyzed archeological materials are pottery, stone (especially obsidian and flint), metal, bone, and other solid material. NAA has proved particularly useful in source and trade route studies, because the trace element concentration in materials such as obsidian can be used as a "fingerprint" to relate an artifact to a source area.

Method. NAA requires obtaining a pulverized 100 mg sample from the object. Samples from pottery are commonly obtained by drilling out a core from the artifact, thereby preserving its outer surface and shape. For small objects such as coins, the analysis requires no sampling.

The samples are crushed, weighed, and sealed in polyethylene or fused silica tubes. The tubes are placed in the core of a specially designed nuclear reactor where the sample reacts with the neutrons in the core. A small percentage of the nuclei of each atom absorbs the neutrons to produce isotopes, some of which are radioactive. After the samples have been irradiated, they are removed from the reactor and placed in the detector. As each radioactive isotope decays, it emits gamma rays of a characteristic energy. These gamma rays are energy-analyzed and counted and the number of counts reflects the concentration of the isotope. Concentration determinations are made by comparing the sample with known quantities of elements that were placed in the reactor at the same time.

Discussion. NAA can be used to analyze for 40 to 76 major, minor, and trace elements. In order to obtain meaningful results for the trace elements, care must be taken to avoid contamination. Sensitivity ranges from less than 0.01 ppm to several percent depending on the element. Accuracy varies for each element but results are generally within 5 to 20 percent of the true value.

Silver and gold can be analyzed with NAA, but extra care is necessary because these elements strongly absorb neutrons. A macroscopic object such as a lump of silver can produce a flux gradient through the object because of absorption in the outer layers.

Advantages. NAA is the dominant and in many cases the only practical method of analysis available for trace elements, including the rare earth elements.

Disadvantages. A major drawback to NAA is that it depends on a nuclear reactor and special detectors and so can be carried out only by specialists at a reactor site. Analysis time can take over a month depending on equipment, accuracy, and elements to be analyzed. Cost per sample is typically \$100 to \$300. Because of this expense, NAA is often reserved for trace elements with major elements being analyzed by atomic absorption or x-ray fluorescence.

Interpretation of Multielemental Data by Advanced Pattern Recognition Methods

The interpretation of data produced by NAA and other forms of elemental analysis is greatly enhanced by the use of statistical pattern recognition methods. Several new computer programs, such as PAM and Masloc, have been produced in the past 5 years which are capable of processing multielemental results and producing easily readable clusters. Both hierarchical and nonhierarchical clustering programs are available, as well as additional programs which take the clusters apart and test whether each object does in fact belong to the group to which it has been assigned.

These advanced pattern recognition methods are particularly useful in provenience studies, where previously unassigned artifacts can be clustered with known source groups

and new clusters identified. A further application is the comparison of visually-based assignments of provenience (those determined by traditional typologies) with assignments determined by NAA or other types of elemental analysis.²⁷

²⁷ P. K. Hopke, *Receptor Modeling in Environmental Chemistry* (John Wiley and Sons, Inc., N.Y., 1985); D. Esse and P. K. Hopke, "Levantine Trade in the Early Bronze Age," *Proceedings of the 24th International Archaeometry Symposium*, J. S. Olin and N. J. Blackman, Eds. (Smithsonian Institution Press, Washington, DC, 1986), pp 327-339.

4 CONCLUSION

This report has reviewed current archeometric technologies in order to familiarize field archeologists with the available methods. The techniques described are powerful and can be used to solve a number of problems. Each technique, however, has drawbacks, and it is important to select those which address specific research questions.

The choice of analytical method employed on archeological materials depends on several factors. The major consideration is the type of information sought. For example, in provenience studies of pottery, choice of method depends on how many elements need to be analyzed for how many samples. Often samples from a large number of sites are required.

Other considerations are the type of equipment available and the relative costs and processing times. In each case, the archeologist must weigh the time and money involved in the sampling and processing against the information desired. It is also wise to consult with the analysts who operate the instruments, since they can often suggest the most cost-effective method and the quickest way to reach the archeologist's objectives. Collaboration between the field archeologist and the instrument specialist is also essential if the results are to be interpreted meaningfully.

Archeometry is an open-ended field, since new applications of all these techniques are still being discovered. The advanced pattern recognition methods currently being developed will continue to enhance the archeological interpretation of multielemental data, particularly in the area of provenience studies.

Finally, most materials can be analyzed by more than one method. Therefore, careful research design is required to apply these instrumental analyses in an efficient, cost-effective manner. For example, the chemical composition of ceramics can be established through NAA, XRF, or ES. The manufacturing techniques of metal artifacts can be revealed through metallography backed with SEM. These options have been discussed in Chapter 3 and are summarized in the decision trees and charts (Figure 1 and Tables 1 and 2) provided in Chapter 2. For further discussion, see Appendices B through D.

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*The following abbreviations are used: AAR - amino acid racemization; AAS - atomic absorption spectroscopy; ES - emission spectroscopy; NAA - neutron activation analysis; PIXE - particle induced x ray emission; SEM - scanning electron microscopy; TL - thermoluminescence; XRD - x ray diffraction; XRF - x ray fluorescence; ESR - electron spin resonance.

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The author points out that the mineral phase of bone (hydroxyapatite) and the major organic phase (collagen) change structure by fluorination and uranium accumulation. Hence assuming a constant dose rate would be erroneous. Ikeya claims to have a computer model to account for changes in dose rate and thus to have produced reliable ESR dates (Ikeya is one of the major figures in this specialty and organized a conference on the subject in Japan in 1985).

Ikeya, M., 1986, "Organic Radicals Detected by ESR in Archaeological Materials," *Abstracts of the 1984 International Archaeometry Symposium*, Smithsonian Institution, Washington, DC, p 67.

He claims to have detected ESR signals from degraded wool, silk, leather, amber, and mummified material. Evidently this is preliminary work.

Szwarcz, H. P., 1986, "ESR Dating of Tooth Enamel," *Abstracts of the 1984 International Archaeometry Symposium*, Smithsonian Institution, Washington, DC, p 124.

He cites advantages of tooth over bone for ESR dating: (1) well-crystallized already; (2) picks up less uranium during burial; (3) organic radical signal background is missing; (4) signal has a long lifetime and high saturation level--favorable for dating of Quaternary specimens.

Skinner, A. F., 1986, "ESR Potential in Aragonite," *Abstracts of the 1984 International Archaeometry Symposium*, Smithsonian Institution, Washington, DC, p 130.

Aragonite--a form of calcium carbonate--is found in shells. TL cannot be used here because heating causes recrystallization to calcite. The ESR signal is usable and persistent, permitting shell samples to be dated back to 300,000 years.

(The following additional papers are important in tracing the development of ESR dating and its specific applications).

Ikeya, M. and Miki, T., 1980, "Electron Spin Resonance Dating of Animal and Human Bones," *Science* 207, pp 977-998.

This study claims to have "demonstrated the reliability" of ESR for dating bone. The signal is associated with the O-radical in hydroxyapatite. Dates between a hundred and a million years ago are claimed to be accessible. Average dose rates of 1 rad/yr in open sites and 0.1 to 0.2 rad/yr in calcite caves are measured.

Hennig, G. J. et al., "ESR Dating of the Fossil Hominid Cranium from Petralona Cave, Greece," 1981, *Nature* 292, pp 533-536.

The calcite deposit on the skull was found to be chemically similar to the top layer on the cave floor. Samples from the skull interior were dated to 200,000 years ago.

Robins, G. V. et al., 1978, "Identification of Ancient Heat Treatment in Flint Artifacts by ESR Spectroscopy," *Nature* 276, p 703.

The ESR signal in unheated geological samples of flint, found to have a g-value of 2.0017, disappeared on heating the specimen to 400 °C. Worked archeological flints exhibited no ESR signal, suggesting heat treatment to improve working properties. Another ESR signal at g = 2.0035 was found in these samples and associated with amorphous (charred) carbon.

Maurer, C., Williams, W. S., Riley, T. J., 1981, "ESR Dating of Archaeological Ceramics: A Progress Report," *MASCA Journal* 1, pp 202-204.

McMorris, D. W., 1971, "Impurity Color Centers in Quartz and Trapped Electron Dating," *Journal of Geophysical Research* 76, pp 7875-7887.

Zeller, E. J., P. M. Levy and P. L. Mattern, "Geologic Dating by Electron Spin Resonance," *Radioactive Dating and Methods of Low-Level Counting* (International Atomic Energy Agency, Vienna 1967), pp 531-540.

METALLOGRAPHY

Betancourt, P. et al., 1978, "Metallurgy at Gournia", Museum Applied Science Center for Archaeology (*MASCA Journal* 1, pp 7-8.

Hodges, H., 1971, *Artifacts*, John Baker Publishers, London, pp 209-219.

**Lechtman, H. N., 1971, "Ancient Methods of Gilding Silver: Examples from the Old and New Worlds," in *Science and Archaeology*, R. H. Brill, ed., Cambridge, MA, M.I.T. Press, pp 2-30.

Gilded silver artifacts from Turkey and northern Peru are compared, revealing different solutions for the production of gold surfaces on silver artifacts in the Old and New worlds.

Maddin, R., Wheeler, T. S., Muhly, J. D., 1980, "Distinguishing Artifacts Made of Native Copper," *Journal of Archaeological Science* 7, pp 211-225.

**Morse, B. F. and Gordon R. B., 1986, "Metallographic Examination of Pre-Columbian Mexican Copper and Silver Artifacts from Mitla, Oaxaca (Mexico)," *Archeomaterials* 1,1, pp 57-67.

Inclusions in copper retain elongation they acquired during cold working, serving as indicators of the total amount of plastic deformation.

PETROGRAPHY

Campling, N. R., 1980, "Identification of Swan River Chert," in *Directions in Manitoba Prehistory*, Winnepeg, Association of Manitoba Archaeologists, pp 291-301.

De Natali, D., 1980, "Petrography, X-ray Diffractometer Analysis and Quarry Sites," *Bulletin of the Massachusetts Archaeological Society* 41, pp 11-14.

Ellis, L., 1980, "Analysis of Cucuteni-Tripolye and Kurgan Pottery and the Implications for Ceramic Technology," *Journal Indo-European Studies* 8, pp 211-230.

Hodges, H., 1971, *Artifacts*, John Baker Publishers, London, pp 190-193, 198.

SCANNING ELECTRON MICROSCOPY (SEM)

Conolly, A., 1976, "Use of Scanning Electron Microscope for Identification of Seeds, with Special Reference to Saxifraga and Papaver," *Folia Quaternaria* 47, pp 29-32.

Fedge, D., 1979, "Scanning Electron Microscopy Analysis of Use-Striae," in B. Hayden, Eds., *Lithic Use-Wear Analysis*, New York, Academic Press, pp 179-87.

Stodulski, L. P., Mailand, H. F., Nauman, D., 1985, "Atomic Emission Spectrographic and Scanning Electron Microscopic/Energy Dispersive X-Ray Studies of European, Middle Eastern, and Oriental Metallic Threads," in *Application of Science to the Dating of Works of Art*, William H. J. Young, Ed., Research Laboratory, Museum of Fine Arts, Boston.

Describes use of SEM to check for gold layers over silver. Energy dispersive x rays are used to determine varying amounts of gold, silver, and copper and to check the composition of corroded layers.

X-RAY DIFFRACTION (XRD)

Isphording, W. C., 1974, "Combined Thermal and X-Ray Diffraction Techniques for Identification of Ceramic Ware Temper and Paste Minerals," *American Antiquity* 39, pp 477-488.

Maggetti, et al., 1984, "Provenance and Technical Studies of Mexican Majolica Using Elemental and Phase Analysis," in *Archaeological Chemistry III* (see above), pp 151-191.

Stout, A.M. and Hurst, A., 1985, "X-Ray Diffraction of Early Iron Age Pottery from Western Norway," *Archaeometry* 27, pp 225-230.

XRD results show (1) pottery never fired as high as 375DoU C, (2) absence of heat-sensitive clay mineral suggests secondary firing of 700-800DoU C, and (3) coloring differences due to firing procedures rather than to use of slip.

EMISSION SPECTROSCOPY

Johnson, W. M. and J. A. Maxwell, 1981, Rock and Mineral Analysis, Vol 27 in *Chemical Analysis: A Series of Monographs on Analytical Chemistry and Its Applications*, P. J., Elving and J. D. Winefordner, Eds., John Wiley and Sons, New York, pp 382-386.

Goffer, pp 40-42 (See General).

Orphanides, G. ,1985, *Radioanalytical Techniques in Archaeology*, pp 22-24.

Rauret, G. and Casassas, E., 1985, "Spectrochemical Analysis of Some Mediaeval Glass Fragments from Catalan Gothic Churches," *Archaeometry* 27, pp 195-201

AAS and ES of two types of window glass, with recent bibliography.

INDUCTIVELY COUPLED PLASMA SPECTROSCOPY (ICP)

Johnson, W. M. and J. A. Maxwell, 1981, Rock and Mineral Analysis, Vol 27 in *Chemical Analysis: A Series of Monographs on Analytical Chemistry and Its Applications*, P. J., Elving and J. D. Winefordner, Eds., John Wiley and Sons, New York, pp 382-386.

Heyworth, M. P., et al., 1986, "The Role of Inductively Coupled Plasma Spectrometry," *Abstracts of the 1986 International Symposium on Archaeometry in Athens*, p 123.

ATOMIC ABSORPTION SPECTROSCOPY (AAS)

Ebdon, L., 1982, *An Introduction to Atomic Absorption Spectroscopy*, Heyden and Son Inc., Philadelphia.

Goffer, pp 51-52 (see General).

Hughes, M. J., Cowell M. R., Craddock, P. T., 1976, "Atomic Absorption in Archaeology," *Archaeometry* 18, pp 19-39.

Tennent, N. H. et al., 1984, "Major, Minor and Trace Element Analysis of Medieval Stained Glass by Flame Atomic Absorption Spectrometry," *Archaeological Chemistry III* (see above), pp 133-150.

With recent bibliography on AAS.

X-RAY FLUORESCENCE SPECTROSCOPY (XRF)

Avaldi, L. et al., 1984, "Quantitative Results of XRF Analyses of Ancient Coins by Monochromatic X-Ray Excitation," *Archaeometry* 26, pp 82-95

Includes bibliography of XRF of coins, jewelry, and pottery.

Brownsword, R. et al., 1984, "X-Ray Fluorescence Analyses of English 13th-16th Century Pewter Flatware," *Archaeometry* 26, pp 237-244.

Hopke, pp 216-218 (energy dispersive XRF) (see General).

Pollard, A. M., Moorey, P. R. S., 1982, "Some Analyses of Middle Assyrian Faience and Related Materials from Tell al Rimah in Iraq," *Archaeometry* 24, pp 45-50.

Energy-dispersive XRF used to determine that copper was the main coloring agent in the alkali glazes.

Yap, C. T. et al., 1985, "Energy-Dispersive X-Ray Fluorescence Analyses of Chinese Porcelains Using AM-241," *Archaeometry* 27, pp 61-63.

PARTICLE INDUCED X-RAY EMISSION (PIXE)

Demortier, G., Terwagne, G., Morciaux, Y., 1986, "Improved Performances for PIXE Analysis of Gold Artifacts," in *Proceedings of the 24th International Archaeometry Symposium*, J. S. Olin and M. J. Blackman, Eds., Smithsonian Institution Press, pp 225-234.

Actual composition of copper, iron, gold, and silver of narrow parts of artifacts can be obtained without sampling.

Ferreira, G. P., Gil, F. B., 1981, "Elemental Analysis of Gold Coins by Particle Induced X-Ray Emission (PIXE)," *Archaeometry* 23, pp 189-198.

Surface composition is found to reproduce bulk composition, at least for high grade gold alloys.

Jacobsen, L., et al., "Elemental Analysis of Fabrics and Glazes of Medieval Majolica from Eastern Spain," *Abstracts for the International Symposium on Archaeometry in Athens*, p 87.

Hopke, pp 218-219 (see General).

Rye, O. S., Duerdon, P. 1982, "Papuan Pottery Sourcing by PIXE: Preliminary Studies," *Archaeometry* 24, pp 59-64.

Certain elements (S, Cl, Br, Zr) may increase their concentration due to impurities in the water used to render clay plastic or in the firing process and should not be included in sourcing studies.

Winter, F. et al., "PIXE Analysis and Late Classical and Hellenistic Imports to Gordion," *Abstracts for the 87th Meeting of the Archaeological Institute of America* (December 1985), pp 7-8.

NEUTRON ACTIVATION ANALYSIS AND INTERPRETATION OF MULTIELEMENTAL DATA BY ADVANCED PATTERN RECOGNITION METHODS

De Atley, et al., 1982, "Comparison of Data Obtained by Neutron Activation and Electron Microprobe Analyses in Ceramics," in *Archaeological Ceramics*, J. S. Olin and A. D. Franklin, eds., Smithsonian Institution Press, Washington, DC, pp 79-87.

See also article by G. Harbottle in same volume, pp 67-77.

**Esse, D. and Hopke, P. K., 1986, "Levantine Trade in the Early Bronze Age," *Proceedings of the 24th International Archaeometry Symposium*, J. S. Olin and M. J. Blackman, Eds., Smithsonian Institution Press, Washington, DC, pp 327-339.

Hancock, R. G. V., 1985, "Neutron Activation Analysis of Ceramics: Problems with Titanium and Calcium," *Archaeometry* 27, pp 94-101.

With bibliography on NAA applied to Roman, Minoan, Mycenaean, Late Bronze Age, Spanish, and Phoenician pottery; medieval floor tiles, and Mexican terra cotta figurines.

Hopke, P. K., et al., 1986, "The Interpretation of Multielemental INAA Data Using Pattern Recognition Methods," *Journal of Radioanalytical and Nuclear Chemistry* (in press).

Kuleff, I., et al., 1985, "Provenance Study of Medieval Bulgarian Glasses by NAA and Cluster Analysis," *Archaeometry* 27, pp 185-194.

Martin, Roger C. , 1986, *Application of Multivariate Statistical Methods to the Analysis of Ancient Turkish Potsherds*, Ph.D thesis, Dept. of Nuclear Engineering, University of Illinois at Urbana-Champaign.

NAA and pattern recognition programs developed by Hopke.

Wisseman, S., Hopke, P. K., Schindler-Kaudelka, E., 1986, "Multielemental and Multivariate Analysis of Italian Terra Sigillata in the World Heritage Museum, University of Illinois at Urbana-Champaign," *Archeomaterials* (in press).

With additional bibliography on NAA of pottery and pattern recognition methods.

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Hall, R., "The Mississippian Heartland and Its Plains Relationships," *Plains Anthropologist*, Vol 12 (1967), pp 175-183.

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GLOSSARY

- absolute dating:** the age of an artifact or event in years before present (bp). In some instances the date can be converted to calendar years (BP).
- accuracy:** the reproducibility and error inherent in a measurement.
- accelerator mass spectrometer 14C dating (AMS):** a new technique that extends the limit of reliable dating to approximately 60,000 years bp. It uses extremely small samples of 0.5 to 5 mg of carbon. It is twice as expensive as standard gas or liquid scintillation counters.
- angstrom:** (Å) one ten-billionth of a meter.
- aplastic inclusions:** all types of non-plastic materials found in clay bodies. Many times they are naturally occurring inclusions in the clay. Those that are purposely added to clays are called temper.
- archeometry:** the measurement of the material properties of an archeological artifact in order to solve problems of chemical composition, technology, chronology, etc.
- authentication:** the establishment of the genuineness of an artifact or work of art by analysis of date, type of materials(s), and/or techniques.
- birefringence:** the refraction of light in two different directions to form two rays.
- chert:** a compact siliceous rock of varying color composed of microorganisms of precipitated silica grains. Occurs as nodules, lenses, or layers in limestone and shales. Its conchoidal fracture has made it highly valued as the raw material for archeological stone tools.
- cold working of metal:** the hammering of metals to produce artifacts. This is a common technique used for the production of artifacts from native copper silver and gold.
- cortex surfaces:** outer layer of lithic nodules that exhibits weathering and abrasion which relates to the material's geological history. In obsidian, the cortex surfaces can identify artifacts which were fashioned from nodules. These surfaces should be avoided when hydration dating is used.
- diet residues:** organic materials left on the surfaces of artifacts which can be analyzed by various techniques to provide information on prehistoric diet. For example, burned starches which are left on the interior surfaces of cooking vessels.
- diffraction:** modification of light in passing by the edges of opaque bodies or through narrow slits or in being reflected from surfaces and in which rays appear to be deflected and produce fringes of parallel light and dark or colored bands.
- diffusion:** reflection of light by a rough reflecting surface, transmission of light through translucent material, or in archeology, the spread of a cultural trait from its point of origin.
- exsitu:** artifacts that have been removed from their original archeological contexts.

"fingerprint": a suite of physico-chemical characteristics of a material that allows it to be distinguished from other materials.

gas chromatography: a process by which complex mixtures of chemical compounds are separated from each other by selective partition between a stationary liquid or solid phase and a mobile gas phase.

grain structure: in cast or worked metals, the periodicity of the crystalline structure is redirected by the development of small "grains"--particles made up of many molecules. The size and shape of these individual grains--the grain structure--convey information about the thermomechanical history of the object.

hardness and microhardness testing: used in metallurgical studies to determine hardness of a metal (Birrell Rockwell scale) by measuring the depression left in a specimen by a small metal ball forced into the specimen for a set time interval at a standard load. Microhardness testing uses a diamond point to make a depression in a single grain of metal which is then measured with a microscope equipped with a calibrated ocular.

hydration rim: the outer layer of obsidian which has combined with water to form perlite. Obsidian is an amorphous glass which is optically isotropic. However, when obsidian combines with water, the hydration rim takes on optical properties different from the parent material. Under a petrographic microscope, this rim can be seen and its thickness measured. The thickness of the hydration rim is a reflection of the time the fresh surface was exposed to the environment. Therefore, when an artifact is fashioned from obsidian, the fresh surfaces represent time zero and begin to hydrate at a constant rate dependent on the ambient temperature.

insitu: artifacts left in their original archeological contexts.

intrasite analysis: the collection of data pertaining to the relationship between members of a single archeological community as seen through their material cultural remains. Intrasite analyses address such questions as the date of the occupation, the definition of activity areas within the site, the spatial organization of the site, the economic organization of the community, subsistence activities, the definition of ritual and ceremonial activities, etc.

intersite analysis: the collection of data pertaining to the relationship between archeological communities as seen through their material cultural remains. Intersite analyses address questions as to the socio-political relationship between sites, the spatial organization of sites on a regional basis, the regional economic relationships, etc.

isotopic fractionation: separation of an element into different isotopes. For example, certain plants have higher concentrations of the heavier isotopes of carbon 13. In carbon-14 dating, it is necessary to establish ratios for ^{12}C , ^{13}C , ^{14}C in order to compare dates obtained from samples of different materials.

isotope: any of two or more species of atoms of an element with the same atomic number and nearly identical chemical behavior but different atomic mass.

lead (Pb) isotope analysis: measures the isotopes of lead found in certain metals such as silver by means of atomic absorption. The ratios of the isotopes of lead in metallic artifacts can be used to correlate with measurements from probable ore sources.

metallography: a form of microscopy which uses light reflected from the polished, etched surface of a sample fragment taken from a metal object. Although metals are crystalline substances, implying a periodic arrangement of atoms, this periodicity is interrupted and redirected in cast or worked metal objects by the development of small "grains"--particles made up of many molecules. The size and shape of these individual grains, which convey information about the thermomechanical history of the object, can be studied using metallography.

micron: one thousandth of a millimeter.

microprobe analysis: also called electron microprobe analysis--is a microanalytical technique which is useful in determining both qualitative and quantitative composition of minute surface areas such as inclusions in ceramics, defects in metal, or paint flecks from a painting. Equipment usually combines a scanning electron microscope, an optical viewing system, and a detector, such as an energy dispersive x-ray detector (EDS or EDX). Increasingly sophisticated microprobes (auger and secondary ion) are now available.

microwear analysis: the study of striations and polish on the edge of lithic tools resulting from their use. Specific uses of lithic tools have been shown to leave characteristic striation and polish patterns which are detectable with the aid of high magnification.

multicomponent site: an archeological site that contains cultural material from more than one archeological phase or assemblage.

"non-destructive": a relative term in archeometry, referring to the extent to which a sample is irretrievably destroyed in preparation. SEM, for example, can be applied to small artifacts of up to one inch, whereas AAS requires a dissolved sample.

obsidian: a naturally occurring glass formed by the rapid cooling of viscous lava. It has conchoidal fracture making it highly valued as a raw material for the production of stone tools.

petrographic microscope: a microscope that uses polarized light and a revolving stage for the analysis of petrographic thin sections.

physico-chemical characterization: the definition of the physical and chemical properties of a material. These data can then be used to describe technological processes involved in the manufacture of the material and/or its provenience.

polarized light: light that has been changed from the ordinary state in which the transverse vibrations occur in all planes passing through the line of propagation to a state in which they are in a single plane.

precision: in materials science, the scale in which a measurement can be taken, i.e., parts per hundred, thousand, million, or billion.

provenience or provenance: the precise source of a raw material (obsidian), the locality where an artifact was manufactured (pottery), or the place where an artifact was recovered in modern times.

radiography: (also x-radiography or x-ray photography) uses x rays to reveal internal structure and technological features of artifacts.

relative dating: age determinations of artifacts or events which allow for their relative chronological ordering but which are not tied to a calendrical system.

resolution: in microscopy, the capability of making distinguishable the individual parts of an object, closely adjacent optical images, or sources of light. Degree of resolution has wide variation from one instrument to another, being lowest with a standard binocular microscope and potential very high with newer scanning electron or transmission electron microscopes.

sensitivity: in analytical techniques refers to the number of elements a particular technique can detect and measure.

sequence: the establishment of a rate of change--in the decay of a radioactive isotope, as in radiocarbon dating or in the thickness of a hydration rim for obsidian hydration dating--correlated with age determinations based on an independently derived dating system. This rate of change can then be used for the dating of artifacts by the calibrated technique. Many techniques are temperature dependent and require different calibration sequences for different geographical areas.

sourcing: in many instances archeological raw materials vary in their trace element chemistry. This provides a basis for the correlation of artifacts with their raw material source locations. These data provide important information on prehistoric procurement of raw materials, trade, and cultural interactions.

spectrograph: a form of spectroscope designed for photographing spectra.

spectroscopy, spectrometry: observation and measurement of portions of the electromagnetic spectrum, usually to characterize a material in terms of chemical composition. There are many types of spectro instruments for recording wavelengths and intensities of gamma rays, x rays, infrared rays, etc.

stable isotope analysis: an analytical technique applied to organic material, especially bone, to determine ratios of different isotopes of the elements carbon and nitrogen to reconstruct diet of early populations. Isotopes of carbon are distributed differently in groups of plants using different photosynthetic pathways. Plants ingested by animals and humans lead to various degrees of enrichment of ^{13}C measurable in bone collagen. Measurement of nitrogen isotopes in bone is used to distinguish between diets of nitrogen-fixing versus non-nitrogen-fixing plants.

"statistical reduction of data": the application of statistical analyses to raw data. For example, multielemental chemical data from a group of artifacts are analyzed to form clusters of individuals which are chemically similar.

stratified site: an archeological site which was occupied periodically for a sufficient time for cultural material to have accumulated representing more than one archeological phase or assemblage. Stratification usually takes the form of

verticle superposition; however, horizontal stratification, i.e., different areas of the site being occupied during different time periods, is also common.

surface analysis: type of analysis which gives the properties of only the surface portion of an artifact. Surface can be defined in terms of angstroms, as in scanning electron microscopy, or microns, as in x-ray fluorescence.

temper: aplastic inclusions purposely added to clay to enhance its workability, decrease shrinkage, reduce thermal shock, and in certain instances to increase porosity.

thermal analysis: measurement of thermal properties such as conductivity, resistance to shock, natural radiation emitted after heating, weightloss after heating, etc., to determine internal structures and reveal processing methods. Techniques applied primarily to the study of ceramics, metals, and stone include differential thermal analysis (DTA), thermal gravimetric analysis (TG), and dating by thermoluminescence (TL).

thin sections: transparent slices of material to be used in microscopic analysis. The object material is cut, mounted on a glass slide, and then ground to the appropriate thickness--for geological specimens, about 30 microns.

transmission electron microscopy (TEM): unlike scanning electron microscopy (SEM) which is primarily a surface analytical technique, TEM provides information on microstructural features inside a specimen by firing electrons through a thin slice. TEM provides higher resolution than other microscopes and higher spatial resolution microanalysis.

typology: in archeology, the classification of artifacts by their form, style of decoration, technique, or other attributes.

x ray: electromagnetic radiations of short wavelengths produced by bombarding metallic targets with electrons that have the property of penetrating solids.

APPENDIX A:

ARCHEOMETRY RESOURCES AND LABORATORIES

The following laboratories or programs are particularly concerned with analyses of archeological materials:

Archeometry Resources

Brookhaven National Laboratory, Upton, New York

Center for Materials Research in Archaeology and Ethnology, Massachusetts Institute of Technology, Cambridge, Mass. (Director: Heather Lechtman)

Conservation Analytical Laboratory, Smithsonian Institution, Washington, D.C. (Archeometrist: Jacqueline S. Olin)

Program on Ancient Technologies and Archaeological Materials, University of Illinois at Urbana-Champaign, 106 Materials Research Laboratory, 104 S. Goodwin, Urbana, IL 61801 (Director: Prof. Wendell S. Williams)

MASCA--Museum Applied Science Center for Archaeology, University of Pennsylvania, Philadelphia, Pa. (Director: Stuart Fleming)

Major Symposia and Proceedings

Application of Science to the Dating of Works of Art, William J. Young (ed.), Proceedings of seminars held in 1978 and 1983 at the Research Laboratory, Museum of Fine Arts, Boston, Mass. (see annotated bibliography for individual articles on TL, SEM, NAA, etc.)

Archaeological Ceramics, Jacqueline S. Olin and Alan D. Franklin, proceedings of seminar on "Ceramics as Archaeological Material," Smithsonian Institution and National Bureau of Standards in 1980, Smithsonian Institution Press (1982)
(Includes introductory section on archeometry, also papers on NAA, Electron Microprobe analysis, SEM, etc.)

International Symposium on Archaeometry (convenes for paper and poster sessions every two years). Publishes both abstracts and proceedings.

1988- to be held in Toronto

1986-Athens, Greece (proceedings in press)

1984-Washington, D.C. Proceedings of the 24th International Archaeometry Symposium, J. S. Olin and N. J. Blackman (editors), Smithsonian Institution Press (1986)

1982- Naples, Italy (Abstracts only)

Journals Dealing With Archeometry

Archaeometry
MASCA Journal
Archeomaterials

Journal of Archaeological Science
Journal of Field Archaeology
Journal of Glass Studies
Society for Archaeological Sciences Newsletter

Laboratories

The following is a partial listing of commercial laboratories organized by analytical techniques. Many of these laboratories perform analyses on a commercial basis for industry, and have had no experience in archeological applications. However, all laboratories listed in this appendix have expressed an interest in analyzing archeological specimens.

The price of analysis and the "turn-around" time for analytical results vary for each laboratory. It is therefore recommended that the appropriate laboratory be contacted and the project be discussed prior to the shipment of samples.

Laboratories (Listed by Analytical Technique)

1. Dendrochronology

Laboratory of Tree-Ring Research

2. Radiocarbon Dating (^{14}C)

Beta Analytic, Inc.

Geochron Laboratories, Inc.

Illinois State Geological Survey

NSF-Arizona Regional Accelerator Facility

Radiocarbon Laboratory, Institute of the Study of Earth and Man, Southern Methodist University

Radiocarbon, Ltd.

Radiocarbon Laboratory, Department of Anthropology, University of California, Riverside (UCR)

Institute of Geophysics and Planetary Physics Radiocarbon Laboratory, University of California at Los Angeles (UCLA)

Scripps Institute of Oceanography

U.S. Geological Survey

3. Amino Acid Racemization (AAR)

Scripps Institute of Oceanography

4. Archeomagnetism

Archaeomagnetic and Paleomagnetic Laboratory

Arkansas Archaeological Survey

Department of Geosciences

Archaeomagnetic Program

5. Thermoluminescence Dating (TL)

Instaar (University of Colorado)

Department of Materials Science and Engineering, University of Washington

6. Electron Spin Resonance (ESR)

Department of Geology, McMaster University, Ontario

7. Obsidian Hydration Dating

Anthropological Studies Center, Sonoma State University

Lithicron

Department of Anthropology, U.C.L.A.

Department of Anthropology, UCR

Department of Anthropology, University of Hawaii

Department of Anthropology, University of Pennsylvania

8. Fission Track Dating

National Bureau of Standards

9. Optical Microscopy

Babcock & Wilcox

Thermo Analytical, Inc.

10. Metallography

Analytical Associates, Inc.

Beckman Industrial

11. Scanning Electron Microscopy (SEM)

Babcock & Wilcox

Beckman Industrial

Center for Microanalysis of Materials

McCrone Associates, Inc.

Thermo Analytical, Inc.

Xerox Analytical Laboratories

12. X-ray Diffraction (XRD)

Babcock & Wilcox

Burgess Analytical Laboratory

Camet Research Inc.

Center for Microanalysis of Materials

CSMRI-Analytica, Inc.

Lambda Research, Inc.

McCrone Associates, Inc.

Spectrochemical Laboratories, Inc.

Xerox Analytical Laboratories

13. Emission Spectrometry (ES)

Analytical Associates, Inc.

Babcock & Wilcox

Beckman Industrial

Rexham National Spectrographic Laboratories

Spectrochemical Laboratories, Inc.

14. Inductively Coupled Plasma Spectroscopy (ICP)

Analytical Associates, Inc.

Babcock & Wilcox

Burgess Analytical Laboratory

CSMRI-Analytica, Inc.

National Spectrographic Laboratories

Spectrochemical Laboratories, Inc.

Thermo Analytical, Inc.

Xerox Analytical Laboratories

15. Atomic Absorption Spectroscopy (AAS)

Accu-Labs Research, Inc.

Analytical Associates, Inc.

Babcock & Wilcox

CSMRI- Analytica, Inc.

Eastern Laboratory Service Association

Multichem Laboratories, Inc.

National Spectrographic Laboratories

Spectrochemical Laboratories, Inc.

16. X-ray Fluorescence (XRF)

Babcock & Wilcox

CSMRI-Analytica, Inc.

National Spectrographic Laboratories

NSF-Arizona Regional Accelerator

Xerox Analytical Laboratories

17. Particle Induced X-ray Emission (PIXE)

Bartol Institute

Center for Microanalysis of Materials

Element Analysis Corporation

NSF-Arizona Regional Accelerator Facility

18. Neutron Activation Analysis (NAA)

General Activation Analysis, Inc.

Illinois State Geological Survey

Thermo Analytical, Inc.

Laboratory Addresses (With Contacts When Available)

Accu-Labs Research, Inc.
11485 W. 48th Avenue
Wheat Ridge, Colorado 80033
Contact: Thomas J. Balka, Marketing
Manager
(303) 423-2766
Technique(s): AAS

Analytical Associates, Inc.
19380 Mt. Elliott
Detroit, MI 48234
Contact: Charles K. Deak CPC
(313) 369-9400
Technique(s): AAS

Anthropological Studies Center
Sonoma State University
Rohnert Park, CA 94928
Contact: Thomas Origer
Technique(s): Obsidian hydration

Archaeomagnetic and Paleomagnetic
Laboratory
Earth Sciences Observatory
University of Oklahoma
Norman, OK 73069
Contact: Dr. Robert L. DuBois, Director
Technique(s): Archeomagnetic dating

Archeomagnetic Program
Laboratory of Public Archaeology
Department of Anthropology
Colorado State University
Fort Collins, CO 80523
Contact: Dr. Jeffery L. Eighmy
Technique(s): Archeomagnetic dating

Arkansas Archaeological Survey
P.O. Box 1249
Fayetteville, AR 72701
Contact: Dr. Daniel Wolfman
Technique(s): Archeomagnetic dating

Babcock & Wilson
Research & Development Division
Lynchburg Research Center
P.O. Box 11165
Lynchburg, VA 24506-0935
Contact: Floyd N. Anderson
(804) 385-3322
Technique(s): Optical microscopy, SEM,
XRD, ES, AAS, ICPS, XRF

Bartol Institute
University of Delaware
Newark, DE 19716
Contact: Professor Charles P. Swann
(302) 451-1279
Technique(s): PIXE

Beckman Industrial
4141 Palm St.
Fullerton, CA 92635
Contact: Dr. John Ling-Fai Wang
(714) 447-2337
Technique(s): Metallography, SEM, XRD,
ICPS

Beta Analytical, Inc.
University Branch
P.O. Box 248113
Coral Gables, FL 33124
Contact: Mr. L. Tamers
(305) 592-7708
Technique(s): Radiocarbon dating

Burgess Analytical Laboratory
525 Ashland St.
P.O. Box 981
North Adams, MA 01247
(413) 663-6769
Technique(s): XRD, gas chromatography,
ICPS

Camet Research, Inc.
318 12th St.
Santa Monica, CA 90402
Contact: Ludwig Keller
(213) 395-4542
Technique(s): Non-routine XRD

Center for Microanalysis of Materials
Materials Research Laboratory
University of Illinois at Urbana-
Champaign
104 S. Goodwin Ave.
Urbana, IL 61801
Analyses for scholarly research by
arrangement only.
Contact: John B. Woodhouse
(217) 333-3888
Technique(s): PIXE, SEM, STEM, TEM,
WDS electron probe.

CSMRI-Analytica, Inc.
5930 McIntyre St.
Golden, CO 80403
Contact: Peter Mihalik, Marketing
Director
(303) 279-2581
Technique(s): XRD, XRF, ICPS, AAS

Dept of Anthropology
University of Hawaii
Honolulu, Hawaii
Technique(s): Hydration dating of
basaltic glass

Department of Anthropology
University of California at Los Angeles
Los Angeles, CA 90024
Contact: Ms. Janet Scalaise
(213) 825-2055
Technique(s): Obsidian hydration

Department of Anthropology
University of California Riverside
Riverside, CA 92507
Contact: Mathew Hall
Technique(s): Obsidian hydration.

Department of Geology
McMaster University
Hamilton, Ontario L8S 4M1 Canada
Contact: H. P. Schwarcz
Technique(s): ESR

Department of Geosciences
University of Arizona
Tucson, AZ 85721
Contact: Dr. Robert Butler
Technique(s): Archeomagnetic dating

Department of Materials Science and
Engineering
Mail Stop FB-10
University of Washington
Seattle, WA 98195
Contact: Professor Tom Stoebe
(206) 543-2600
Technique(s): TL dating

Eastern Laboratory Service Association
517 North George St.
York, PA 17404
Contact: Donald E. Nickol
(717) 846-4953
Technique(s): AAS

Element Analysis Corporation (EAC)
1696 Capitol Circle S.W.
Tallahassee, FL 32304
Contact: Jim Spearing
(904) 576-5115
Technique(s): PIXE

ESA Laboratories Inc. (ESAL)
43 Wiggins Avenue
Bedford, MA 01730
Contact: Dr. Reginald M. Griffin
(617) 275-0100
Technique(s): gas chromatography, mass
spectroscopy, ion chromatography,
liquid chromatography and various
wet chemical procedures.

General Activation Analysis, Inc.
11575 Sorrento Valley Road #214
San Diego, CA 92121
Contact: Lawrence E. Kovar
(619) 755-5121
Technique(s): NAA

Geochron Laboratories, Inc.
24 Blackstone Street
Cambridge, MA 02139
Technique(s): Radiocarbon dating

Illinois State Geological Survey
State Geological Survey Division
Natural Resources Building
615 East Peabody Dr.
Champaign, IL 61820
Contact: Barry W. Fisher
(217) 344-1481
Technique(s): Radiocarbon dating

Instaar
Campus Box 450
University of Colorado
Boulder, CO 80309
Contact: Dr. Steven Forman
(303) 492-1196
Technique(s): TL dating

Institute of Geophysics and Planetary
Physics
University of California at Los Angeles
Los Angeles, CA 90024
Technique(s): Radiocarbon dating

Isotec, Inc.
Analytical Services Dept.
3858 Benner Road
Miamisburg, OH 45342
Contact: Mark Lemley
1-800-448-9760
Technique(s): Stable isotope analysis

Laboratory of Tree-Ring Research
University of Arizona
Tucson, Arizona 85721
Contact: Dr. Jeffery Dean
(602) 621-2320
Technique(s): Dendrochronology

Lambda Research, Inc.
1111 Harrison Ave.
Cincinnati, OH 45214
(513) 621-3933
Technique(s): XRD

Lithichron
716 Procter Court
Woodland, CA 95695
Contact: Robert Jackson
(916) 666-1754
Technique(s): Obsidian hydration dating

McCrone Associates Inc.
850 Tasquinelli Dr.
Westmont, IL 60559
Contact: Dr. Mark Germani
(312) 887-7100
Technique(s): Optical microscopy, XRD,
SEM, TEM, electron microprobe,
gas chromatography, secondary ion
mass spectrometry

Department of Anthropology
University of Pennsylvania
Philadelphia, PA
Contact: Chris Stevenson
Technique(s): Obsidian hydration dating

MultiChem Laboratories, Inc.
171 Lincoln Street
Lowell, MA 01851
Contact: Michael G. Gardell
(617) 459-9172
Technique(s): AAS

National Bureau of Standards
Gaithersburg, MD 20899
Contact: Steve Carpenter

NSF-Arizona Regional Accelerator
Facility, PAS (Bldg. 81)
University of Arizona
Tucson, AZ 85721
Contact: J.O. Stoner Jr.
Technique(s): AMS radiocarbon dating,
PIXE, XRF

Radiocarbon Laboratory
Department of Anthropology
University of California Riverside
Riverside, CA 92507
Technique(s): Radiocarbon dating

Radiocarbon Laboratory
Institute for the Study of Earth and Man
Southern Methodist University
Dallas, TX 75222
Contact: Herbert Haas
Technique(s): Radiocarbon dating

Radiocarbon, Ltd.
Route 2, Box 21E
Lampasas, TX 76550
Technique(s): Radiocarbon dating

Rexham National Spectrographic
Laboratories
P.O. Box 31480
Cleveland, OH 44131
Contact: E.A. Mestancik
(216) 447-1550
Technique(s): ES, ICPS, AAS,

Robert Jackson
State Office of Historic Preservation
Box 942896
Sacramento, CA 94296-0001
(916) 322-9062 (office)
(916) 666-1754 (home)
Technique(s): Obsidian hydration

Scripps Institute of Oceanography
University of California, San Diego
La Jolla, CA 92037
Contact: Dr. H.E. Suess
Technique(s): Radiocarbon dating, AAR
dating

Spectrochemical Laboratories, Inc.
8350 Frankstown Ave.
Pittsburgh, PA 15221
Contact: Tom Fulton
(412) 371-2345
Technique(s): ES, X-ray spectroscopy,
XRD, ICPS, AAS

Structure Probe
P.O. Box 656
West Chester, PA 19381
Contact: Charles Garber
(800) 242-4SPI(4774)
Technique(s): STEM, SEM, electron
probe, TEM, auger electron spectro-
scopy.

Thermo Analytical, Inc. (TMA)
160 Taylor Street
P. O. Box 2360
Monrovia, CA 91016-2360
Contact: John D. McCarthy
(818) 357-3247
Technique(s): Optical Microscopy, SEM,
ICPS, NAA

Xerox Analytical Laboratories
Xerox Corporation 0114-42D
Joseph C. Wilson Center for Technology
Rochester, NY 14644
Contact: Judy A. Coene
(716) 422-3675

Technique(s): Gas chromatography, mass
spectroscopy, electron microprobe
analysis, ICPS, auger electron spec-
trometry, XRD, XRF, TEM, SEM

U.S. Geological Survey
Radiocarbon Laboratory
National Center 971
Reston, VA 22092
Technique(s): Radiocarbon dating

APPENDIX B:

PROVENIENCE OF SELECTED MIDDLE MISSISSIPPIAN VESSEL FORMS FROM THE CENTRAL MISSISSIPPI VALLEY USING HIERARCHICAL AND NONHIERARCHICAL CLUSTERING OF NAA RESULTS

T. J. Riley,¹ P. Hopke,² R. Martin,² and J. W. Porter,¹ Departments of ¹Anthropology, ²Nuclear Engineering and Institute for Environmental Studies, and Program on Ancient Technologies and Archaeological Materials, University of Illinois, Urbana, Illinois, 61801.

Abstract

Sherds representing four ceramic types from a time period ca. A.D. 1150 to 1400 in the Central Mississippi Valley, U.S.A. were subjected to petrographic analysis and neutron activation analysis (NAA). Twenty-six trace elements were identified and both hierarchical and nonhierarchical cluster analyses were used to segregate the sherds by locality. Assumptions of differential clay histories in the three localities underlay the attempt. Where necessary, the results of NAA were weighed against petrographic analysis to determine reasons for cluster failure. Distance separation in both hierarchical and nonhierarchical clustering was reasonable. When clusters were broken down by type, hierarchical clustering was good. Results suggested that stimulus diffusion through exchange of people or ideas about ceramic style was the main cause of similarities within types. Trade in finished wares is not indicated.

Introduction

Four different ceramic wares from sites in different parts of the Central Mississippi Valley were chosen to attempt provenience studies using petrographic analysis of thin sections, NAA, and concomitant hierarchical and nonhierarchical clustering techniques on the activation data. The wares included waterbottles, plates, beakers and a cordmarked jar form from a series of related phases of village horticulturalist settlements dating from ca. A.D. 1150 to 1400.

Methods and Materials

A total of 140 sherds from the four wares were chosen from 16 sites from three localities (Figures B1 through B4) which exhibit different characteristics of siltclays. All the samples were subjected to neutron activation analysis NAA, but only 126 sherds were complete enough to be thinly sectioned for petrographic analysis. Locality 1, represented by 26 sherds from three sites, is located on the Kaskaskia River, a westward flowing tributary of the Mississippi. Locality 2 is represented by 90 sherds from 10 sites on the east side of the Mississippi above the confluence with the Kaskaskia, while Locality 3, represented by 24 sherds from 3 sites, is located on the east side of the Mississippi River below its confluence with the Ohio. All the samples chosen for the study were from Middle Mississippian contexts dating from ca. A.D. 1150 to 1400. Three of the four wares, the beaker, plate and waterbottle forms, have a wide distribution in the Central Mississippi Valley from ca. A.D. 1250 to 1400, though their occurrence at any particular site is restricted. The fourth ware, called in Illinois the Cahokia cordmarked jar, has a restricted occurrence in Illinois, Wisconsin, and along the Mississippi River in Missouri, and dates from ca. A.D. 1150 to 1400. It is quite abundant at sites where it is found, and

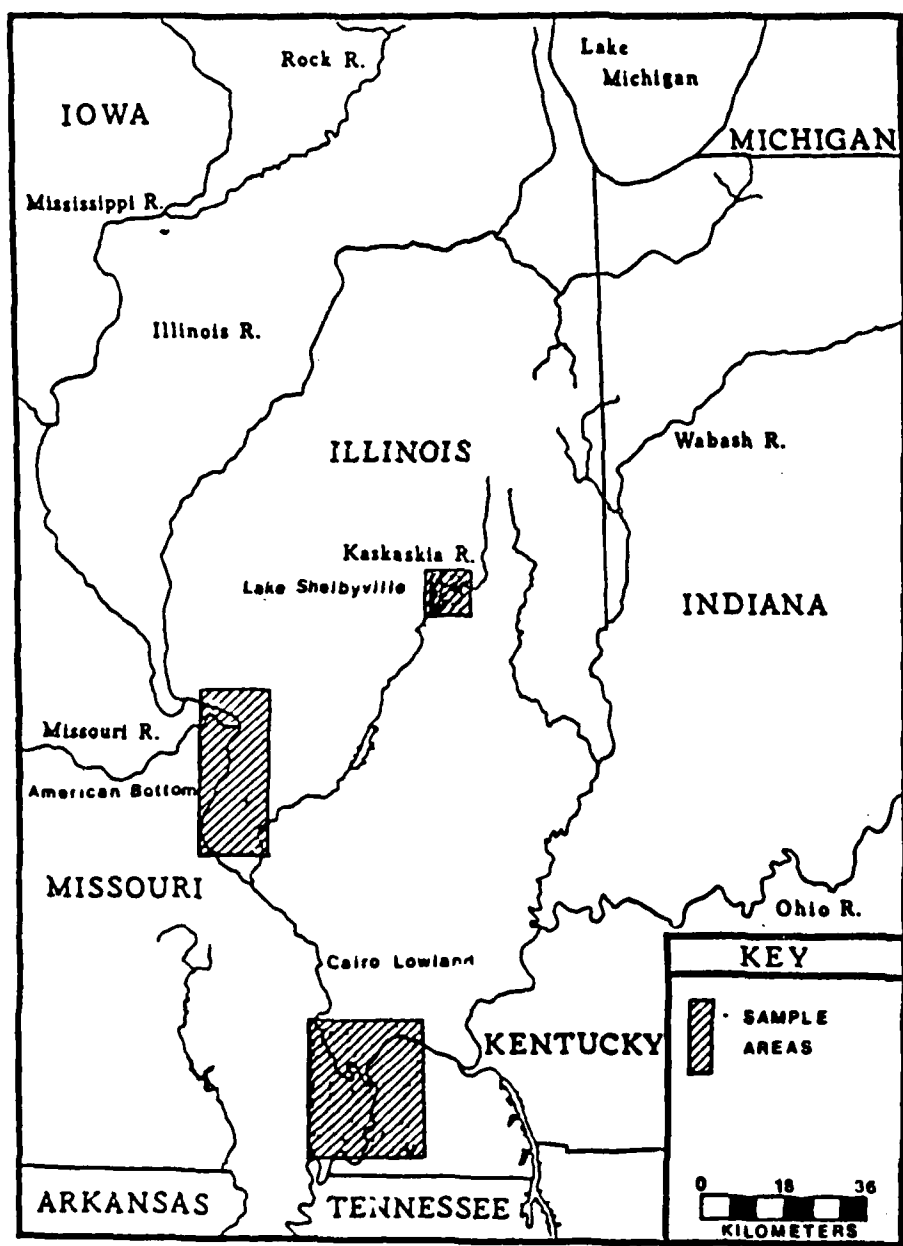


Figure B1. General location of Mississippi Valley sample sites.

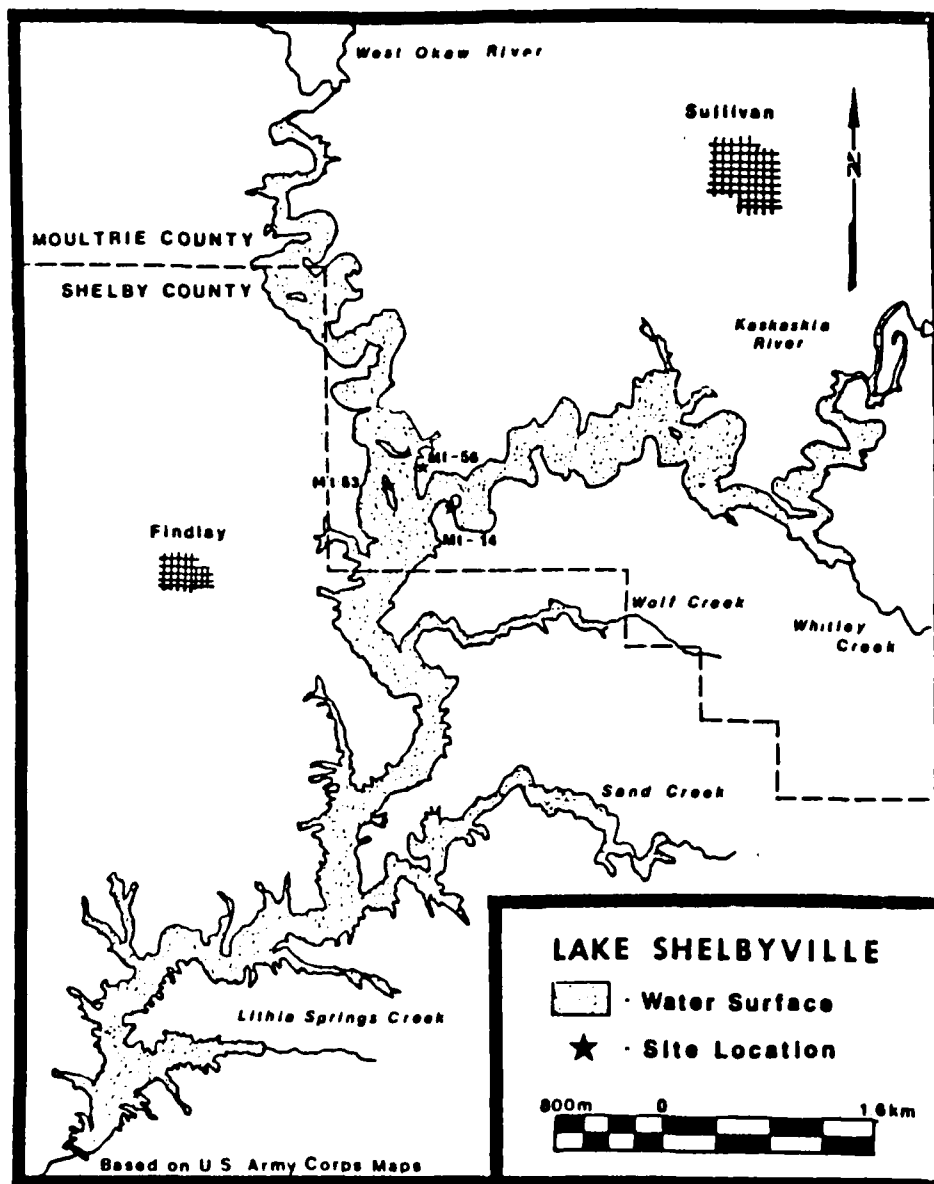


Figure B2. Locality 1, Kaskaskia River at Lake Shelbyville.

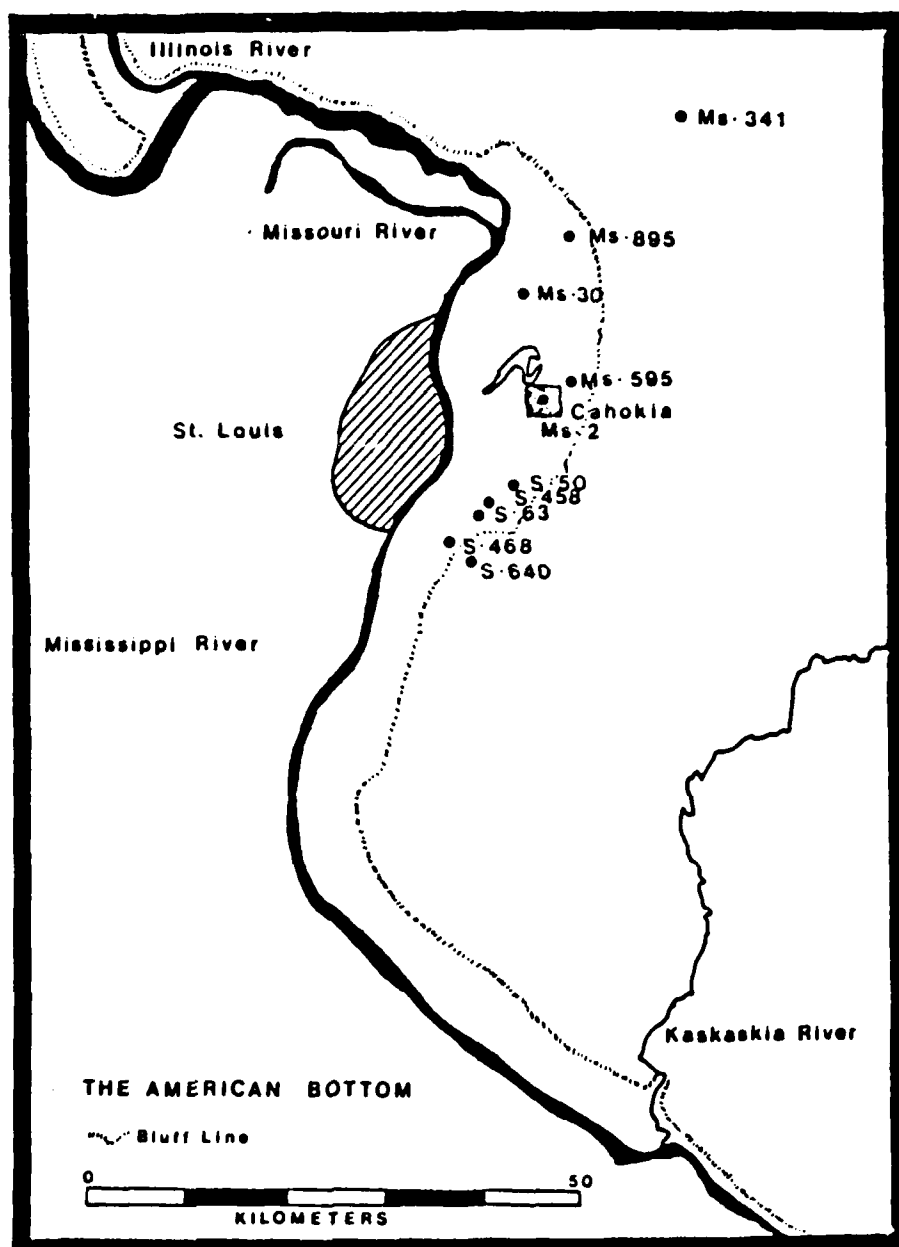


Figure B3. Locality 2, Mississippi above Kaskaskia River—the American Bottom. (Adapted from *American Bottom Archeology*, C. Bareis and J. W. Porter, Eds. [University of Illinois Press, 1984], p 5.)

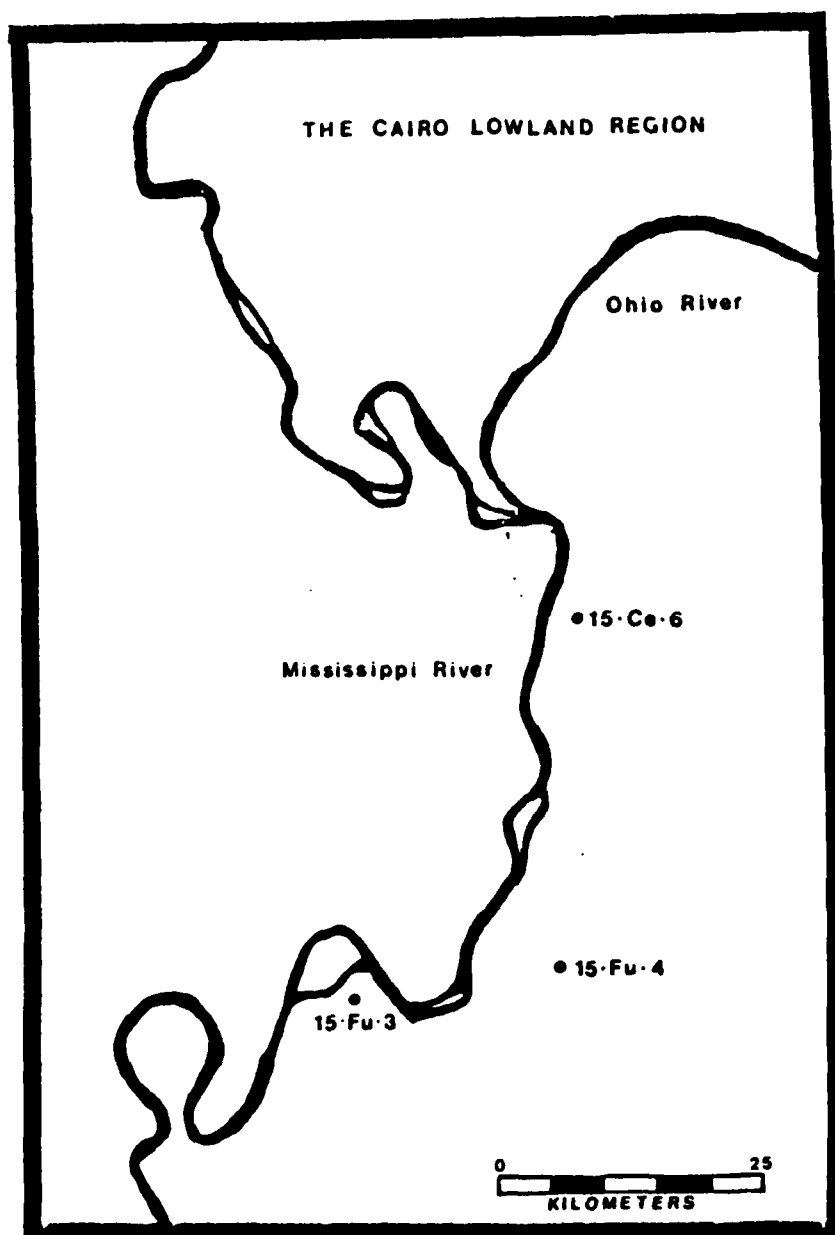


Figure B4. Locality 3, Mississippi River below Ohio River—Cairo Lowland Region.

is probably a utilitarian ware.²⁸ The first three wares are suspected by some researchers to have been spread as the result of trade. The Cahokia cordmarked jar, on the other hand, is often thought to be a locally-made ware. The research reported here was designed to determine whether the sherds from the three localities were similar enough to suggest an exchange of finished goods from one of the provinces to another, or whether they would cluster well within each locality, suggesting the local manufacture of the ware.

Assumptions

The assumptions governing the direction of the research were fairly simple. If the siltclays represented in the various parts of the Mississippi influenced by other river systems were different from one another as Grim²⁹ suggests, then locally made ceramics from each locality should produce trace element arrays that would be more similar to one another than to the ceramics from other localities. Unlike some research conducted earlier,³⁰ no attempt was made to identify sources of raw clay from each locality. Raw clay has not undergone the same treatment as the clay in manufactured vessels. It has not been mixed, tempered or fired. When raw clay is treated by the modern researcher, there is little control over the similarity of the end product to the prehistoric template. For these reasons, it was decided to treat the finished vessels as the units of study and the trace elements revealed by NAA as the variables that should cluster for each locality.

Thin Sections

One hundred twenty-six sherds were visually inspected, photographed, and thinly sectioned. Sections were examined under transmitted light conditions at magnifications up to 400X. Less attention was paid in the analysis to mineral phases present than to tempering material, sand/silt fraction, and the presence of biogenic opal (indicative of an alluvial backwater mud). In addition to the sections, clay samples were kept for x-ray diffraction, although only a small sample of the sherds was subjected to this analysis (which is not reported on here). The thin sections, under petrographic analysis, permitted the gross analysis of sherds that did not cluster as expected by locality. The thin section samples are separated into tables by vessel type (Tables B1 through B5). The following abbreviations are used.

On these tables the sample number indicates state, vessel type, and river valley (see legend). More details regarding the types of temper encountered in thin sections can be found in Porter.³¹ The abbreviations are given in the legend below.

²⁸ Charles R. Moffet, *The Mississippian Occupation of the Upper Kaskaskia Valley: Problems in Culture History and Economic Organization*, unpublished P.H.D. Thesis (Department of Anthropology, University of Illinois at Urbana-Champaign, 1986).

²⁹ Ralph Grim, *Clay Mineralogy* (University of Illinois Press, Urbana, IL, 1957).

³⁰ M. Abacskal, R. G. Harbottle, and E. V. Sayre, "Correlation Between Terra Cotta Figurines and Pottery From the Valley of Mexico and Source Clays by Activation Analysis," in *Archaeological Chemistry*, Curt Beck, Ed. (American Chemical Society, Washington, DC, 1974).

³¹ James W. Porter, "Thin Section Analysis of Ceramics," *The Robinson's Lake Site*, George R. Milner, Ed., FAI 270, Site Report, Vol 10 (American Bottom Archaeology, 1984), pp 133-170.

Table B1
Abbreviations in Data Tables

Column		Legend
Sample number	State	I = Illinois K = Kentucky
	Vessel type	C = Cahokia Cordmarked Jars P = Plates B = Beakers W = Bottles
	River valley	Ks = Kaskaskia Mi = Mississippi
Temper		Sh = Shell Gg = Grog Bo = Bone
Grain Size		VC = Very coarse (2.0 - 1.0 mm) C = Coarse (1.0 - 0.5 mm) M = Medium (0.5 - 0.25 mm) F = Fine (0.25 - 0.125 mm) VF = Very Fine (0.125 - 0.062 mm)
Silt		H = High (40 - 30%) M = Medium (30 - 10%) L = Low (10 - 0%)
Biogenic opal		C = Common P = Present

Table B2
Sample Data—Water Bottles

Sample	Site	Temper	Temper Size (mm)	% Temper	Grain Size	N (of Grains)	Silt	Biogen Opal	Comments on Temper in the Grog. Frag.
IWKs-7A	MT-53	Sh	2.2x0.13		M-F	6	M	P	
IWKs-7B	Mt-53	Sh	1.2x0.14		M-F	10	M	P	
IWMI-55	S-50	Sh	1.3x0.19		V-F	6	L	C	
IWMI-67	S-63	Sh	1.4x0.06		F	6	M	P	
IWMI-73	S-63	Sh	1.0x0.06		F-VF	11	L	P	
IWMI-82	Ms-2/3	Sh	2.2x0.13		M-F	2	L	P	
IWMI-91	Ms-2/3	Sh	2.1x0.13		F-VF	6	L	C	
IWMI-93	Ms-2/3	Sh	3.2x0.51		M-VF	6	M	P	
IWMI-108	S-468	Sh	1.1x0.19	25-30	F-VF	8	L	P	
IWMI-115	S-468	Gg	(1.5x0.13) 2.4 (0.8x0.06)	40-45	VF	1	H	P	Sh, some grog is isotropic

Table B3
Sample Data—Beakers

Sample	Site	Temper	Temper Size (mm)	% Temper	Grain Size	N (of Grains)	Silt	Biogen Opal	Comments on Temper in the Grog. Frag.
IBKs-1	Mt-53	Sh	1.0x0.70			C-F	3	L	C
IBMI-56	S-50	Gg	1.2			VF	9	H	P
IBMI-57	S-50	Sh	2.9x0.13			F-VF	9	H	C
IBMI-58	Ms-595	Sh	1.3x0.19			M-VF	6	L	P
IBMI-59	Ms-595	Sh	0.6x0.13			VF	6	L	P
IBMI-60	Ms-595	Sh	0.8x0.13			VF	4	L	P
IBMI-61	S-63	Sh	1.2x0.13			VF	3	L	P
IBMI-68	S-63	Sh	0.9x0.28			M-VF	6	L	P
IBMI-69	S-63	Sh	1.1x0.13	25-30	F-VF	4	L	P	
IBMI-88	Ms2/3	Gg	1.3	30-35	F	6	L	P	Sh, Fine crushed
IBMI-116	S-468	Sh & Bo	(1.8x0.64) (0.3x0.14)		M-F	8	M	P	

Table B4
Sample Data—Plates

Sample	Site	Temper	Temper Size (mm)	% Temper	Grain Size	N (of Grains)	Silt	Biogen Opal	Comments on Temper in the Grog. Frag.
IPKS-2	MT-56	Sh	2.9x0.20		C-M	8	M	P	
IPKS-3	MT-53	Sh	1.8x0.26		M-VF	9	M	M	
IPKS-6	MT-53	Sh	1.9x0.64		F-VF	6	M	P	
IPKS-7	MT-53	Sh	1.9x0.19		M-F	6	M	P	
IPKS-8	MT-53	Sh	2.6x0.26		M	3	L	C	Poor slide
IPMI-71	S-63	Sh	1.3x0.13		F	6	L	P	
IPMI-74	S-63	Sh	1.3x0.32	10	M	13	M	P	
IPMI-84	Ms-2/3	Sh	2.2x0.06	34.5	F-VF	6	M	P	
IPMI-85	Ms-2/3	Gg	0.96		F-VF	8	L	C	Sh + another type unit
IPMI-86	Ms-2/3	Gg	1.50		M-F	8	L	P	Sh
IPMI-87	Ms-2/3	Sh	1.3x0.60		M-F	25	L	P	
IPMI-89	Ms-2/3	Gg	0.96		F	5	M	C	Sh, 5% grog
IPMI-90	Ms-2/3	Sh	2.2x0.45		F	2	L	P	

Table B4 (cont'd)

Sample	Site	Temper Temper	Temper Size (mm)	% Temper	Grain Size	N (of Grains)	Silt	Comments on Temper Biogen in the Opal Grog. Frag.
IPMi-92	Ms-2/3	Gg	0.96		F	6	M	C Sh
IPMi-94	Ms-2/3	Gg	1.20		F	6	M	PSh + another type?
IPMi-95	Ms-2/3	Sh	1.8x0.26		F-VF	8	M	C
IPMi-96	Ms-2/3	Sh	2.3x0.38	30-35	F	7	M	P
IPMi-98	Ms-2/3	Sh	2.3x0.38	30-35	F	7	M	P
IPMi-103	Ms-2/3	Sh	1.8x0.13		F	5	L	C
IPMi-104	MS-2/3	Gg	0.96		F	6	M	P Sh
IPMi-105	MS-2/3	Sh	2.6x0.13		F	6	M	P
IPMi-106	MS-2/3	Gg	1.00		F	6	M	P Sh, Ident. to 94.
IPMi-107	S-468	Sh	0.3x0.45		VF	5	L	C
IPMi-114	S-458	Sh	1.7x0.13		VF	5	L	C
KPMi-121	Fu-4	Gg	3.2		F-VF	5	M	C Sh + Gg.
KPMi-122	Fu-4	Gg	2.6				M	P Sh + Bo, Gg.
KPMi-123	Fu-4	Gg	2.1				M	P Sh + untemp?
KPMi-124	Fu-4	Gg	1.6	30-35	M-F	6	L	P Sh (diff. types)
KPMi-125	Fu-4	Gg	1.0	5-10	M-F	6	H	P Sh Gg
KPMi-126	Fu-4	Gg	1.4	10	F-VF	6	H	P Sh
KPMi-127	Fu-4	Gg	1.7		C-VF	3	H	C Sh + untemp?
KPMi-128	Fu-4	Gg	1.3	15	F-VF	6	M	P Sh + untemp?
KPMi-129	Fu-3	Gg	1.3		M-F	7	M	P Sh
KPMi-130	Fu-3	Gg	1.6	30	M-F	6	M	P Sh + untemp?
KPMi-131	Fu-3	Gg	1.3	30	M-F	9	M	C Sh + untemp?
KPMi-132	Fu-3	Gg	1.3		M-F	5	M	C Sh + untemp?
KPMi-133	Fu-3	Gg	2.2		F	4	H	C Sh + untemp?
KPMi-134	Fu-3	Gg	1.8	20	M-F	7	M	C Sh + untemp?
KPMi-135	Fu-3	Gg	1.1		C-M	8	M	C Sh
KPMi-136	Fu-3	Gg	1.9		F	5	H	C Sh

Table B5

Sample Data - Cahokia Cordmarked Jars

Sample	Site	Temper	Temper Size (mm)	% Temper	Grain Size	N (of Grains)	Silt	Biogen Opal	Comments on Temper in the Grog. Frag.
ICKs-1	Mt-14	Sh	3.5x0.19	22	VF	6	M	P	
ICKs-2	Mt-14	Sh	3.3x0.26	33	C	8	M	P	
ICKs-4	Mt-53	Sh	4.2x0.96		C-M	17	M	P	
ICKs-5	Mt-53	Sh	3.5x0.64	39	M-VF	5	L	P	
ICKs-8	Mt-53	Sh	7.7x0.60	34	F	5	L	P	
ICKs-9	Mt-53	Sh	4.5x0.19	31	M-VF	4	L	P	
ICKs-10	Mt-56	Sh	2.4x0.45	29	C-M	8	M	P	
ICKs-11	Mt-56	Sh	3.8x0.13	30	C-F	6	M	C	
ICKs-12	Mt-56	Sh	2.6x0.13	31	M-F	7	M	P	
ICKs-13	Mt-56	Sh	2.6x0.26	18	C-M	4	M	P	
ICKs-14	Mt-56	Sh	3.8x0.26	23	C-M	7	M	P	
ICKs-15	Mt-56	Sh	3.0x0.19	24	M	4	M	P	
ICKs-16	Mt-56	Sh	5.1x0.77		VC-M	9	M	P	
ICKs-17	Mt-56	Sh	2.6x0.19		M	5	M	P	
ICKs-18	Mt-56	Sh	5.1x0.64		C-M	7	L	P	
ICKs-19	Mt-53	Sh	3.5x0.30		M-F	4	M	C	
ICKs-20	Mt-14	Sh	2.2x0.19		M	4	H	C	
ICMI-30	Ms-895	Sh	1.2x0.19	15	M	8	H	P	
ICMI-31	Ms-895	Sh	3.5x0.50		M-F	3	M	P	
ICMI-32	Ms-895	Sh	1.6x0.13		F-V	4	L	P	
ICMI-33	Ms-895	Sh	1.8x0.13		VF	5	M	P	
ICMI-34	Ms-895	Sh	1.7x0.26		VF	5	M	P	
ICMI-35	Ms-895	Sh	1.3x0.19		F-VF	4	L	P	
ICMI-36	Ms-895	Sh	0.9x0.13		M	6	L	P	
ICMI-37	Ms-895	Sh	1.2x0.06		F-VF	5	L	C	
ICMI-38	Ms-895	Sh	3.2x0.50		F	5	L	P	
ICMI-39	Ms-895	Sh	2.4x0.38		F	6	L	P	
ICMI-40	Ms-895	Sh	1.8x0.13		M-F	6	L	C	
ICMI-41	Ms-895	Sh	3.5x0.50	25.3	VF	6	L	C	

Table B5 (cont'd)

Sample	Site	Temper Temper Size (mm)	% Temper	Grain Size	N (of Grains)	Silt	Biogen Opal	Comments on Temper in the Grog. Frag.
ICMi-42	Ms-895	Sh	1.5x0.13	F	4	L	P	
ICMi-43	Ms-895	Sh	3.8x0.13	VF	5	L	P	
ICMi-44	Ms-895	Sh	4.2x0.19	VF	6	L	P	
ICMi-45	Ms-895	Sh	2.9x0.19	VF	3	L	P	
ICMi-46	Ms-895	Sh	1.8x0.50	26.7 VF	3	L	P	
ICMi-47	Ms-895	Sh	2.9x0.96	VF	3	L	P	
ICMi-48	Ms-895	Sh	1.9x0.26	F-VF	5	L	P	
ICMi-50	Ms-895	Sh	1.7x0.32	F-VF	8	L	P	
ICMi-51	Ms-895	Sh	2.6x0.19	F-VF	5	L	P	
ICMi-52	Ms-895	Sh	1.7x0.13	F	5	M	P	
ICMi-53	Ms-895	Sh	1.8x0.13	F	3	M	P	
ICMi-54	MS-895	Sh	1.6x0.13	M-F	5	M	P	
ICMi-62	?	Sh	2.1x0.13	M-F	4	M	C	
ICMi-63	S-63	Sh	2.9x0.13	M-F	7	M	C	
ICMi-64	S-63	Sh	2.6x0.19	F-VF	6	H	C	
ICMi-65	S-63	Sh	1.6x0.26	F-VF	5	L	C	
ICMi-66	S-63	Sh	1.4x0.26	F-VF	6	M	P	
ICMi-70	S-63	Sh	2.4x0.19	23.4 F	3	M	P	
ICMi-71	Ms-895		2.1x0.19	21.0 F	5	L	C	
ICMi-72B	S-63	Sh	4.2x0.32	F	5	M	P	
ICMi-72A	Ms-341	Sh	2.6x0.13	M-F	10	H	P	
ICMi-73	Ms-895	Sh	2.4x0.32	M-VF	8	M	C	
ICMi-94	Ms-895	Gg	2.6	M-VF	6	M	C	Sh, sandy paste
ICMi-75	Ms-341	Sh	2.6x0.19	F	8	H	P	
ICMi-80	S-468	Sh	2.6x0.38	F-VF	5	L	P	
ICMi-99	?	Sh	2.9x0.13	M-VF	5	M	P	
ICMi-100	Ms-2/3	Sh	2.2x0.19	F-VF	6	M	P	
ICMi-101	Ms-2/3	Sh	2.9x0.19	M-VF	8	M	P	
ICMi-102	Ms-2/3	Gg	4.0	M-F	4	L	P	Sh, similar to CMI-100
ICMi-117	S-468	Sh	1.9x0.06	M-VF	5	M	P	

Following the temper type is a column for temper size. There is a great deal of variation in this category. The table only provides the measurements of the largest fragments observed. In the case of grog, the longest dimension is given. For shell, the largest, rectangular fragments are selected and the measurements given in the column. Measurements for shell fragments in grog-tempered samples are usually not provided since with the second crushing of the shell it is usually much smaller and, therefore, not characteristic of the sherd.

The percent of temper is indicated in some cases. Exact figures were extracted from a point count technique and ranges (e.g., 40 to 45 percent in IWMi-115) from visual estimates. A set of test tiles (thin sections) with known amounts of temper were used for practice.

The sand content is given in terms of grain size with the largest grain being measured in each thin section. Because the sherds are studied as thin sections (30 microns thick) only some of the grains will actually be cut through a plane providing the true dimension. Therefore, in this study the size of grains is expressed in the tables as abbreviated Wentworth scale items. The range is provided, with N being the number of grains measured. This form of presentation was chosen due to the limited amount of area available for study with only one thin section per vessel.

The silt content was estimated after a point count was made of a few typical sherds to represent high, medium, and low.

The presence of biogenic opal was noted under high power (400X), although in some instances a few large fragments could be observed at 100X. The notation C (for common) indicates that in each field chosen at random a few or more fragments of biogenic opal were observed. The letter P (present) indicates that some opal was noted but usually after a few different fields of view were inspected. In a few thick slides it was difficult to see the biogenic opal.

The last column is used to record observations regarding the temper of the grog fragments.

Neutron Activation

The neutron activation analyses were conducted on 140 samples in the same manner as described by Wisseman, et al.³² The analysis procedure sought values for 37 elements. However, it was found that for 11 elements there was a large fraction of the samples for which the values were below the limits of detection. Hence, the statistical analyses were performed on the concentrations of 26 elements in the 140 objects.

Clustering

Hierarchical agglomerative clustering was carried out using the program AGCLUS which takes the matrix of dissimilarity values from the program DSTCMP, and clusters the individual sherds using any one of seven criteria. The process is initiated by calculating a matrix of similarity or dissimilarity values between each pair of objects, after a z-transform is done converting the data for each variable to a mean of zero and a

³²S. V. Wisseman, P. K. Hopke, and E. Schindler-Kaudelka, "Multielemental and Multivariate Analysis of Italian Terra Sigillata in the World Heritage Museum, University of Illinois at Urbana-Champaign," *Archaeomaterials*, Vol 1 (1987), pp 101-117.

standard deviation of one. These values represent a quantification of the relative closeness of objects within n-dimensional space. Although many combinations of dissimilarity measures and clustering criteria are possible, a squared Euclidean distance method was used for distance calculations, followed by a clustering based on a simple mean within cluster technique. This combination represents the cluster size as the mean squared distance of all the points as measured from the centroid of the cluster, and the clustering criterion emphasizes a tight cluster. This result can be interpreted as the cluster size measuring the within-cluster variance, and the clustering minimizing this variance.

Nonhierarchical clustering was carried out using MASLOC³³ and the graphical representation of results called silhouettes.³⁴ MASLOC is a cluster analysis technique based on the p-median model of location theory.³⁵ It permits a clustering, for each value of p, of the given objects into p groups, with each group being provided with a representative object.³⁶ Another program, PAM, employs the same clustering algorithms and displays the results using silhouettes (graphical representations of the results of nonhierarchical clustering). The silhouettes are also a tool for interpreting the robustness of clusters. The results presented here exhibit the clustering of 19 representative objects from 140 cases. The technique is described by Rousseeuw.³⁷

Results

Petrographic Analysis

A summary of the results of the petrographic analysis suggests that, with a few exceptions, all the wares noted were manufactured locally at their respective sites. Judging from the lack of vitrification, and the variety of oxidized and reduced surfaces, the vessels were uniformly fired at temperatures between 550 and 700 °C, for short periods in open pit firings. Vessel paste in all specimens appears to have been derived from backswamp muds from floodplains, as indicated by varying amounts of biogenic opal. The vessel temper was, in most cases, shell, with 27 samples exhibiting "grog" temper (the inclusion of crushed pottery sherds as a tempering material). In all cases where "grog" temper was noted, shell was also present in the tempering material. In several samples of plates from locality 3, the "grog" itself was found with and without shell tempering. This indicates that waster pot sherds were being recycled as temper for new ceramics again and again in this locality. A beaker from a site in locality 2 showed evidence of bone being used as a tempering material, while a plate from locality 3 exhibited both shell and bone temper.

³³D. L. Massart, F. Plastria, and L. Kaufman, "Nonhierarchical Clustering with MASLOC," *Pattern Recognition*, Vol 16, No. 5 (1983), pp 507-516.

³⁴P. J. Rousseeuw, "Silhouettes: A Graphical Aid to the Interpretation and Validation of Cluster Analysis," *Reports of the Department of Mathematics and Informatics*, No. 84-14 (Delft University of Technology, Delft, The Netherlands, 1984).

³⁵H. D. Vinad, "Integer Programming and the Theory of Grouping," *Journal of the American Statistical Association*, Vol 64 (1969), p 506-519.

³⁶Rousseeuw, p 507.

³⁷Rousseeuw.

Hierarchical Clustering

The results of the hierarchical clusterings are presented in Figures B5 through B9. The brackets cluster samples by similarity; as the brackets move to the right the clustered items are less similar. The clusterings exhibit a good separation of wares by locality when the sample is divided by ware as it is in Figures B6 through B9. Less separation is noted when the sample is taken as a whole (Figure B5), although several distinct clusters are noted. Using the total sample undifferentiated by ware, the hierarchical clustering technique appears to relate clusters from different localities to one another, when in fact the separations are clear at different levels of the hierarchy.

The problem with agglomerative hierarchical clustering is that small clusters are combined to form fewer, larger cluster. Thus, the clustering at each stage depends on the results of the previous stage and hence early errors in classification cannot be rectified as the clusters become larger and their characteristics better defined.

Nonhierarchical Clustering

The nonhierarchical technique, as represented in the silhouettes, does not suffer from the superficial relationships noted in the hierarchical display. It also permits the visual determination of the strength of the cluster, the nearest neighboring cluster to each case within a given cluster, and the strength of the relationships of the cases to each other within the cluster. This last item is represented by a value of $s(i)$, where s identifies the cluster and i indicates the closeness of the relationships within the cluster (higher values of i show tighter clustering). The results are thus much easier to interpret than those of the hierarchical clustering, and it is easy to determine the weakest cases in each cluster, those which could as easily belong to the nearest neighbor cluster. Several clusters stand out in terms of their width as being stronger than the others, notably clusters 18(.40), 4(.37), 17(.35), 12(.27) and 15(.25). Cluster 18, however, is an isolate with only two cases represented in the object.

While the clustering is not perfect at 19 representative objects, it is not too bad, although the average silhouette is only .11. It might be suggested that a number of the cases be removed in the basis of doubt about their placement within a certain cluster. However, there is a definite separation of the majority of cases into clusters suggestive of locality-dependent clusters. In other words, the clustering appears to be natural in most instances. A silhouette breakdown into specific wares has not been attempted yet, but it is expected that the clusters will be stronger and silhouette widths greater with finer control over the wares chosen as cases.

Conclusions

In conclusion, both the hierarchical and non-hierarchical techniques used here exhibit clustering that can be associated with the locality from which the ceramics were collected. The few anomalies remain to be examined using other techniques, but in general ceramic manufacture appears to be a locality dependent process in the Central Mississippi Valley at the period from ca. A.D. 1150 to 1400, even where rare forms such as plates, beakers and water bottles are concerned. While a few examples may represent trade, the majority of the artifacts appear to have been locally manufactured, suggesting a transfer of people or procedural and stylistic modes rather than the transfer of ceramics in trade.

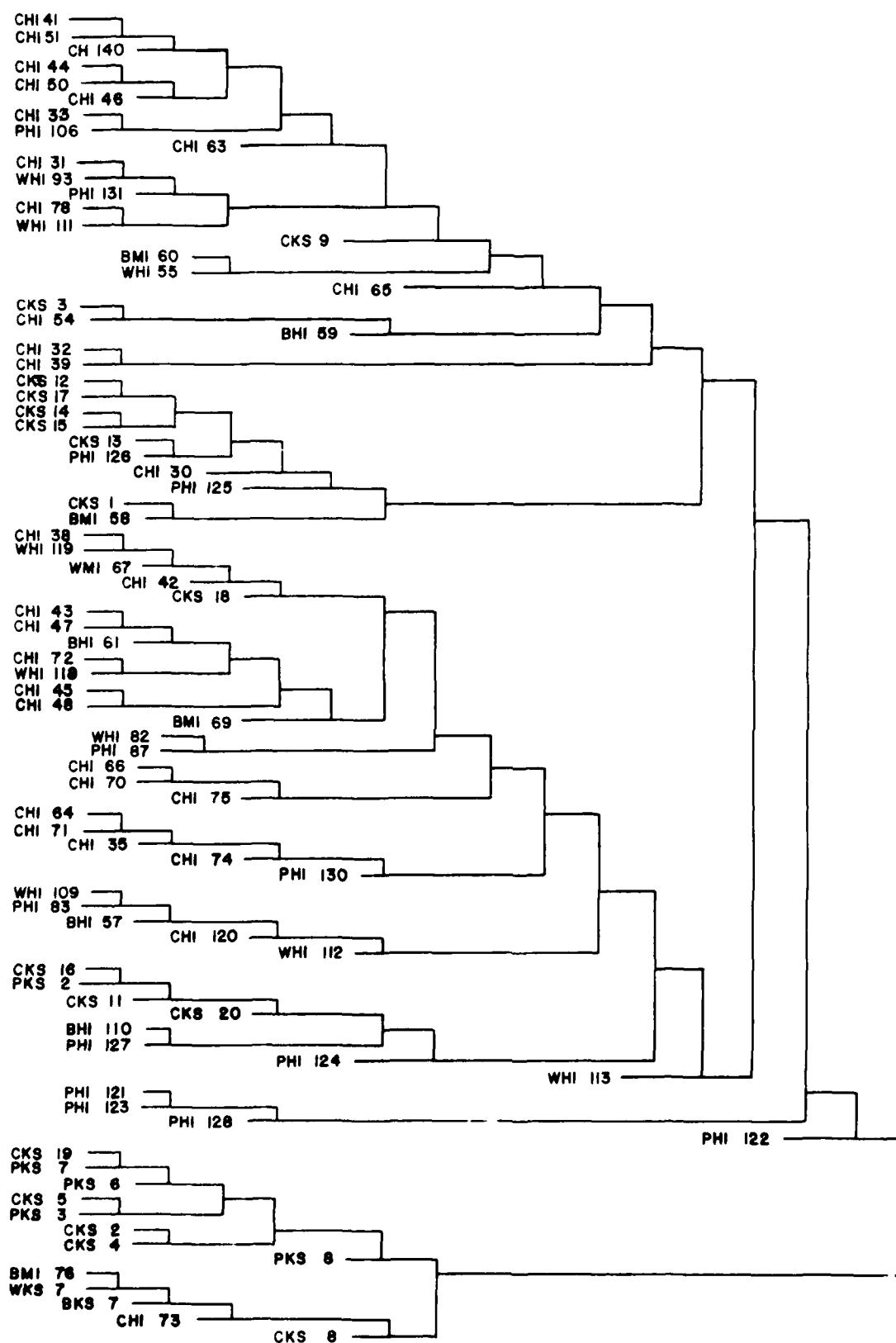


Figure B5. Cluster, 86 samples, 26 variables.

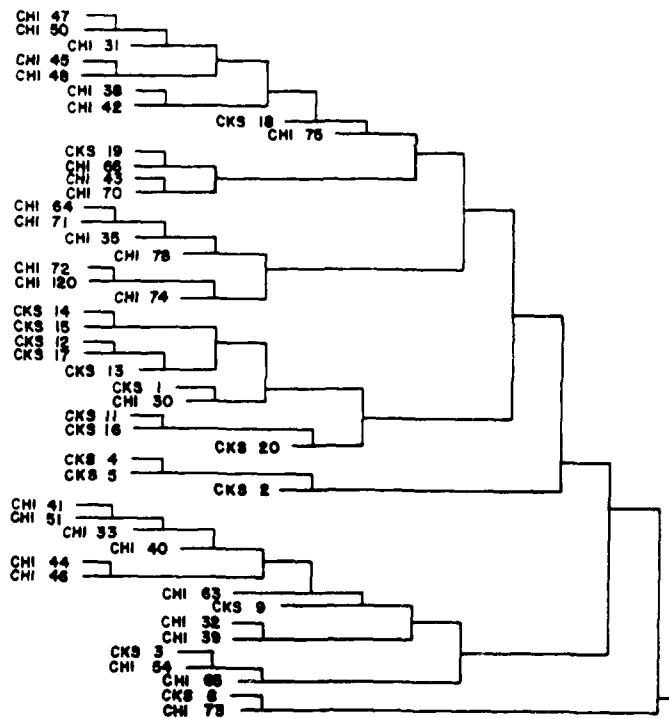


Figure B6. Cahokia cordmarked, 18 samples, 26 variables.

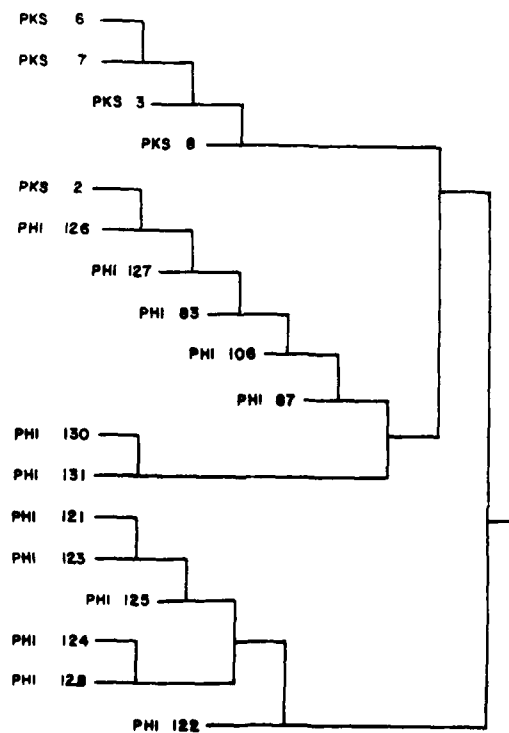


Figure B7. Plates, 18 samples, 26 variables.

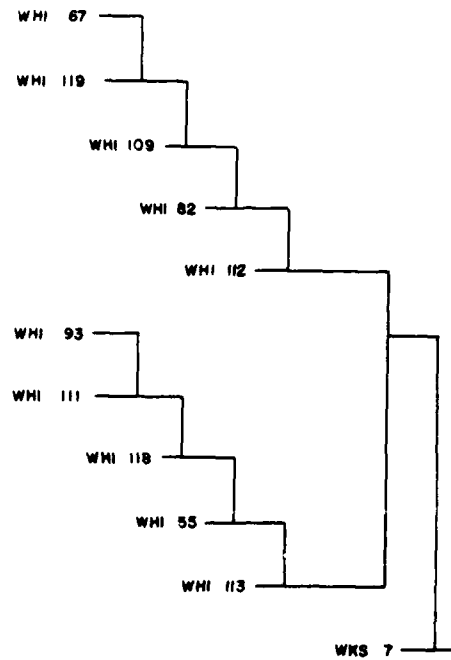


Figure B8. Water bottles, 11 samples, 26 variables.

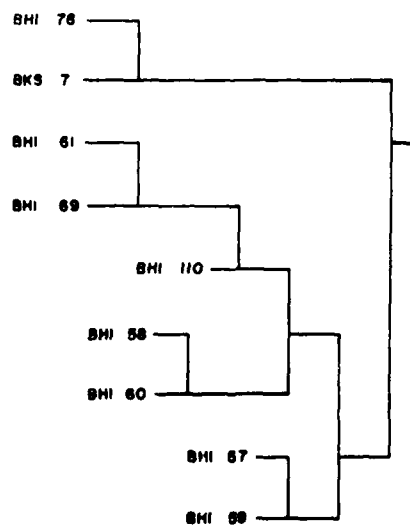


Figure B9. Beakers, 9 samples, 26 variables.

The concept of major Mississippian Site Unit Intrusions (SUIs) across the Mississippian Heartland has dominated thinking about this particular cultural tradition for over a hundred years. Hall³⁸ and others have questioned this interpretation of Mississippian culture for Aztlan and for the major Georgia Mississippian site at Ocmulgee. While there is little doubt that there was a widespread exchange in certain objects of copper, marine shell, and in chert materials used in the manufacture of hoes, the distribution across Mississippian societies of other traits such as those symbols that have been characterized as the Southern Cult could have occurred through diffusion rather than by direct trade or exchange of items. This would be consonant with the pattern that is discernible in the ceramic materials reported on here. Both the apparent utilitarian forms such as the Cahokia cordmarked jar and the plates, and the beaker and bottle forms which appear to be more specialized, are apparently local productions, despite the fact that they share a majority of manufacturing techniques and formal stylistic elements.

If we can rule out the exchange of these items in any great numbers in the period from ca. A.D. 1200 to 1450, then we must posit either an exchange of persons responsible for ceramic manufacture, either in alliance relationships such as marriage, the transfer of people in a slave trade related to enmities between groups or in trade of slaves after warfare or raiding, or the transmission of the ideas of ceramic manufacture from one polity to another during seasonal or coincidental contact. Either way, we can conclude that ceramics were not a major part of the long distance trade that occurred in some exotic items during Mississippian times.

³⁸Robert Hall, "The Mississippian Heartland and Its Plains Relationships," *Plains Anthropologist*, Vol 12, No. 36 (1967), pp 175-183.

APPENDIX C:

MULTIELEMENTAL AND MULTIVARIATE ANALYSIS OF ROMAN TERRA SIGILLATA IN THE WORLD HERITAGE MUSEUM, UNIVERSITY OF ILLINOIS*

The project is a study of a museum collection of Roman pottery acquired through a dealer in the early 1900's. The original source was possibly the dredging of the Tiber river in Rome in the 1890's.

The following summary and accompanying decision tree, Figure C1, provide another example of how to apply archeometric techniques to archeological materials.

Normally archeometric techniques would not be applied to such a small group of artifacts from an uncertain source. However, in the case of Roman terra sigillata pottery, manufacturer's and potter's stamps give a great deal of information which is not available on unstamped pottery. Stamps allow the archeologist to assign specific manufacturing centers in cases where manufacturers are known from literary sources.

First, 117 sherds were visually examined and classified according to color, type of surface treatment, and manufacturer's and potter's stamps. Areas of manufacture were tentatively identified as Northern Italy, Arezzo, and Central Italy.

Since most of the sherds are from unspecified sites, it was decided to attempt elemental analysis of the clay types in order to distinguish different manufacturing centers. NAA was chosen because it produces the large suite of major, minor, and trace elements needed to discriminate between visually similar clays.

Of the 117 sherds, 36 remained unclassified after visual examination; these were selected for NAA. They were sherds with unknown or illegible stamps and non-Arretine fabrics as well as several sherds with stamps of the manufacturer Gellius.

Multivariate analysis of the NAA data was performed to aid in the identification of groups based on similar clay composition and to test NAA results against those obtained by visual examination. Agglomerative hierarchical clustering was the primary analysis technique. In addition, three other analyses were performed as cross-checks on the agglomerative technique: divisive hierarchical clustering, MASLOC, and principal components analysis. Clusters from these analyses were identified and then the membership of each artifact in each group was tested by SIMCA. Six major clusters were identified, indicating at least four manufacturing centers.

*S. Wisseman, P. Hopke, E. Schindler-Kaudelka.

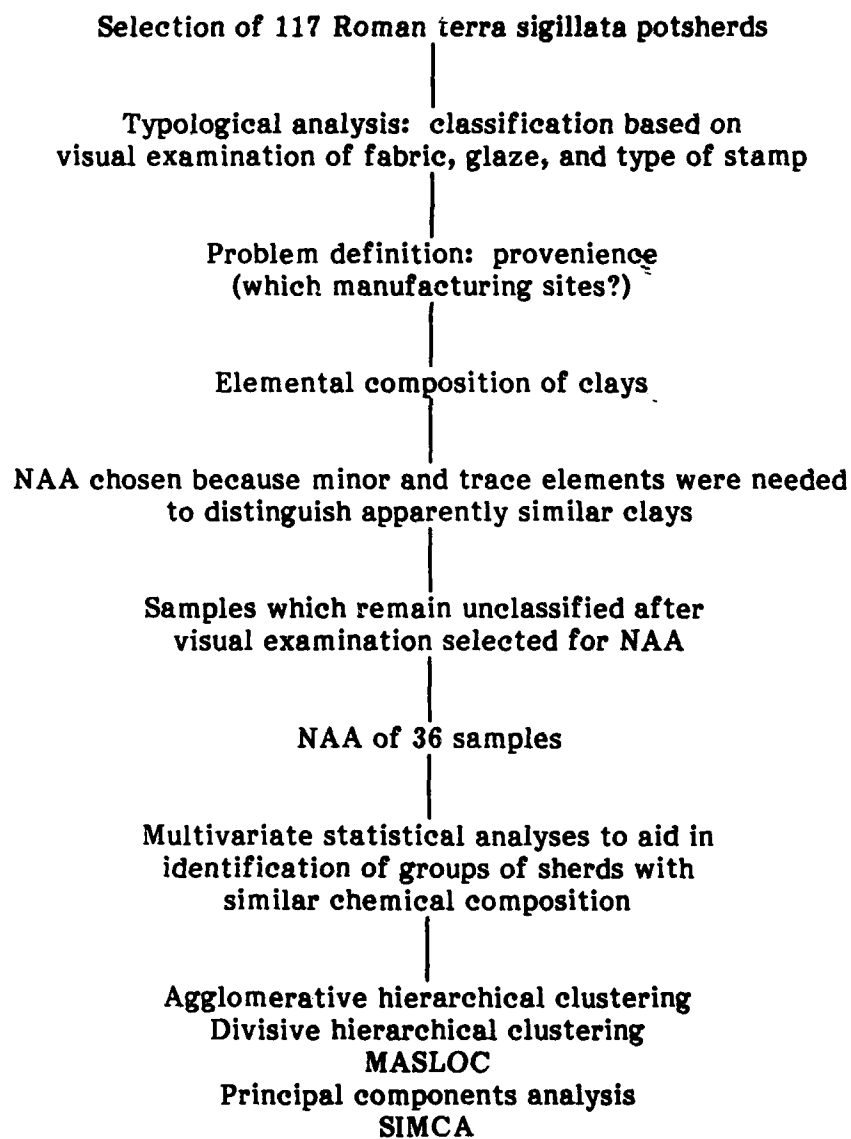


Figure C1. Decision tree for Roman pottery analysis.

APPENDIX D:

ECUADORIAN VOLCANIC ASH CORRELATION STUDY*

Successful application of advanced techniques to archeological problems is the responsibility of the archeologist. It depends on control of the archeological context of the materials, research design, and the ability to communicate with laboratories conducting the analyses.

The number of highly technical analyses in materials research which can be applied to artifacts and fossils has increased enormously. The number of measurements which can be performed on a single artifact is almost limitless. Therefore, the archaeologist must understand which questions are relevant to these technical analyses and will produce results bearing directly on a research problem. For example, prior to submitting radiocarbon samples to the laboratory, the context of the samples, the sample size, and the archeological event which is being dated must be considered to insure the correct selection of samples and correct interpretation of results.

Designing a research strategy which is hierarchically organized is an essential first step. Research strategies should employ simple and direct methods of analysis (usually less expensive) prior to investing in more technical and expensive instrumental techniques. This strategy accomplishes two things: (1) data from the preliminary analysis can be used in interpreting results produced by the more technical analyses discussed in this manual; and (2) research questions become more focused at each level of the hierarchy, so fewer analyses are required to resolve a research problem.

An example of this type of research design was recently employed in the correlation study of volcanic ash (tephra) mantles which buried archeological sites in Ecuador, South America.³⁹ Samples of tephra were collected from a number of possible source volcanoes in the vicinity of the study area, and compared to tephra samples recovered from archeological excavations at two contemporary sites (one located in the Andean highlands, and one located in the tropical rainforest, west of the Andes Mountains). At the lowland site, two archeological occupations were defined, one dating to 1000 B.C. and the other to A.D. 1200 to 1660. Both of these occupations were terminated by volcanic eruptions; the earlier eruption occurred at 350 B.C. and the later eruption at A.D. 1660.

The purposes of the analysis were (1) to investigate the possibility that the earlier occupation at both sites was terminated by the same volcanic eruption, and (2) to identify the volcanic source responsible for the eruption. Additionally, the possibility was investigated that the later occupation at the lowland site was terminated by a well documented historic eruption in A.D. 1660.

*J. Isaacson.

³⁹J. Isaacson, *Volcanic Activity and Human Occupation of the Northern Andes: The Application of Tephrostratigraphic Techniques to the Problems of Human Settlement in the Western Montana During the Ecuadorian Formative*, unpublished Ph.D thesis (Department of Anthropology, University of Illinois at Urbana-Champaign, 1987).

The research strategy was designed as follows (a corresponding decision tree is shown in Figure D1):

1. Lithological examination of all tephra samples with low powered magnification.
2. Mineralogical analysis of ferromagnesian mineral suites under polarized light. Mineral suites were isolated by dense liquid separation.
3. Comparison of pumice clast thin sections with groupings formed by the mineralogical examination.
4. EDS microprobe analysis of magnetite phenocrysts extracted from tephra samples.
5. X-ray fluorescence and neutron activation analysis of bulk pumice samples for trace element correlations study of tephra samples.

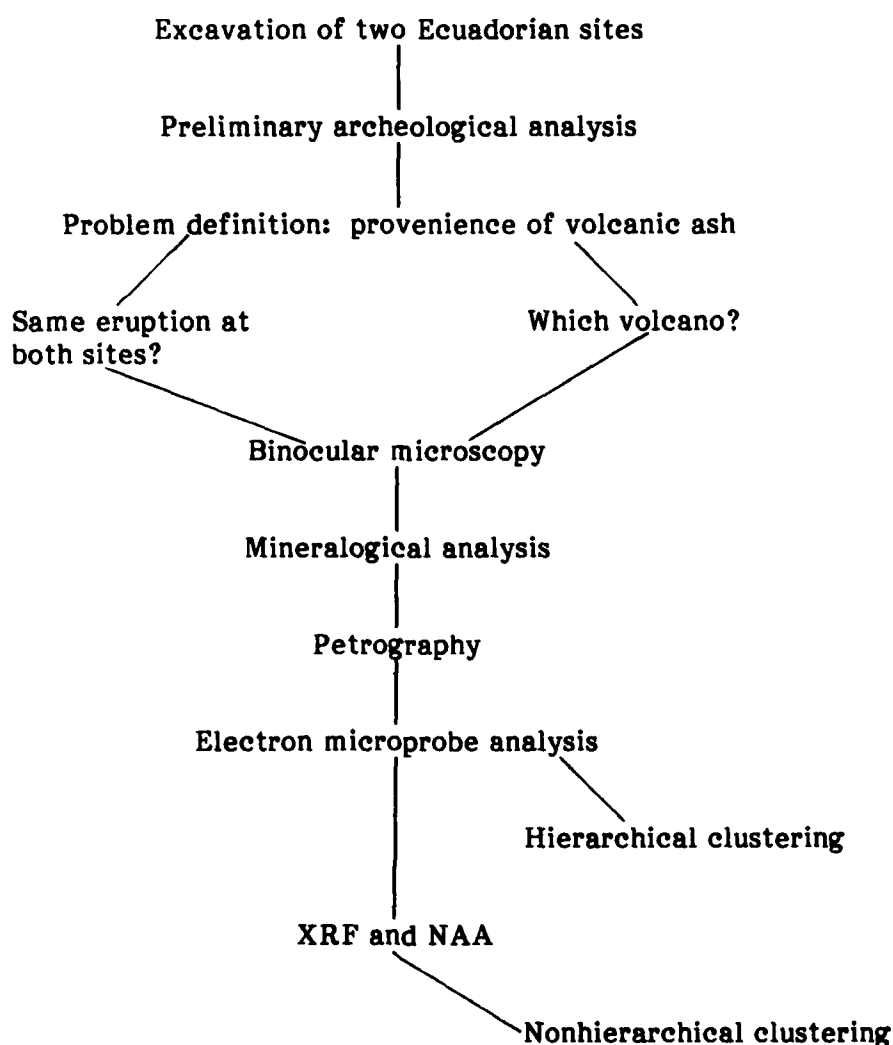


Figure D1. Decision tree for correlation study of volcanic ash mantles.

The first analysis was conducted with low power magnification to compare the lithology of the samples. This was accomplished by observing samples under a binocular microscope at 40X magnification, and grouping samples according to similarities and differences in appearance and phenocryst inclusions. This analysis eliminated two possible source volcanoes from the study since their lithology was obviously different from other samples. This analysis proved inexpensive and effective for sorting and eliminating potential sources.

The second step in the analysis was separating the crystal inclusions in the tephra samples by means of dense liquid separation techniques and comparing crystal inclusions (phenocrysts) from each sample. This concentrated the number of phenocrysts and enhanced the recognition of differences between samples. The process was time-consuming and more expensive than step one, but the major investment was time.

At this stage in the study, large pumice clasts were extracted from the tephra samples and thin sectioned. The sectioned clasts were compared under polarized light. Groupings of tephra samples with their probable sources were established based on the presence or absence of key minerals in the samples. Although more expensive than the first level of the analysis because it involved the use of separation funnels, chemical supplies, production of thin sections, and access to a petrographic microscope, this procedure was relatively inexpensive. Reliable data for the sourcing of the A.D. 1660 deposit at the lowland site were obtained and the possible sources of the earlier tephra deposit were narrowed.

The next analysis involved the elemental analysis of magnetite crystals in the tephra samples by means of an electron microprobe, to identify the volcano responsible for the remaining unsourced tephra mantle. Included in this stage of the study were samples from the A.D. 1660 volcanic source and samples from the lowland site. These samples were included as an independent check of the electron microprobe results.

Results of this analysis in the form of elemental concentrations were clustered through the application of multivariate statistical analysis (Euclidian squared distance hierarchical clustering).

Clustering analyses produce groupings at various degrees of similarity expressed in numerical values. In hierarchical clustering, all samples eventually are subsumed under a single cluster. The investigator, in this case the archaeologist, judges at what degree of similarity the clusters represent "real" groupings of the data, not an artifact of the statistical algorithm. The validity of this judgement is enhanced by results of previous analyses. Without the previous results, distinguishing "real" from "artificial" groupings would be difficult.

The resultant clusters grouped the A.D. 1660 volcanic source pumice with the correct tephra deposit, substantiating the correlation based on the mineralogical analysis. Two possible source volcanoes were also grouped with the earlier tephra deposit at both the highland and lowland sites. Thus the number of possible volcanic sources for the remaining tephra mantle were reduced to two possibilities. The hypothesis that both archeological sites were buried by the same eruption was also confirmed.

This level of analysis was considerably more expensive than the previous two studies. However, the total cost of the analysis was significantly reduced by the elimination of a number of potential volcanic sources based on the lithology and mineralogy of the tephra samples.

The final analysis in the correlation study attempted to discriminate the volcanic source responsible for the 350 B.C. tephra mantle which devastated the highland and lowland archeological sites. X-ray fluorescence and neutron activation analysis were applied to bulk pumice samples from the two possible volcanic sources and to the tephra samples from the two sites. Results of this analysis were analyzed using a nonhierarchical clustering algorithm to form silhouettes of the data.

Unfortunately, it was not possible to identify which of the two volcanoes was responsible for the tephra at the two sites, based on the silhouettes. However, the analysis did provide valuable information about the eruption sequence suggesting that the source volcano was located to the east of the lowland site. This information allowed the identification of the Volcano Pululagua as the source for the tephra, with its final eruption occurring at 350 B.C.

The purpose of this example is to illustrate the necessity of designing a research strategy which will provide a number of independent results for use in evaluating the progressively more intricate, and costly, analyses. At the same time, the number of analyses necessary at each step are reduced, thus decreasing the overall cost.

ABBREVIATIONS

AAR	amino acid racemization
AAS	atomic absorption spectroscopy
AF	alternating field
AMS	accelerator mass spectrometer
B.P.	before present
CRM	chemical remnant magnetism
EPR	electron paramagnetic resonance (same as ESR)
ES	emission spectroscopy
ESR	electron spin resonance
F _A	ancient (magnetic) field
F _L	laboratory (magnetic) field
ICP	inductively coupled plasma spectroscopy
IRM	isothermal remnant magnetism
NAA	neutron activation analysis
NBS	National Bureau of Standards
NRM	natural remnant magnetism
PIXE	particle-induced x-ray emission
RYBP	radiocarbon years before present
SEM	scanning electron microscope
SQUID	superconducting quantum interference device
TL	thermoluminescence
TRM	thermal remnant magnetism
VRM	viscous remnant magnetism
XRD	x-ray diffraction
XRF	x-ray fluorescence

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